

SURFACE ANALYSIS STUDIES OF Nb₃Sn THIN FILMS*

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Abstract

A recent study to optimise the coating of thin-film Nb₃Sn cavities has resulted in coating procedures that can fabricate 1.3 GHz cavities capable of reproducibly achieving fields of >16 MV/m with record high Q s $>1 \times 10^{10}$ at 4.2 K. However, the performance of these next generation SRF cavities is as yet well below the theoretical maximum performance expected of Nb₃Sn, thus giving ample room for further improvement. Recent measurements strongly suggest that the current limitations are due to local defects and irregularities in the coated surface. In this paper we analyse the surface of both sample coupons and cavity cut-outs, with a view to identifying and understanding the origin of surface non-uniformities that could lead to increased surface resistance and cavity quench.

INTRODUCTION

The A15 superconductor Nb₃Sn has shown considerable promise in replacing niobium as the material of choice for superconducting radio-frequency cavities. Although initially plagued by issues of Q -slope, single-cell 1.3 GHz ILC-style cavities coated at Cornell university have demonstrated, repeatedly, fields of up to 16 MV/m with Q 's of $\approx 10^{10}$ at 4.2 K [1]. However, these cavities still fall considerably short of Nb₃Sn's theoretical limits, particularly in terms of accelerating gradient.

Concurrent to the studies on the RF performance of Nb₃Sn cavities, significant efforts to analyse the properties of the Nb₃Sn film directly have been undertaken. In this paper, we discuss the presence of thin (on the order of the penetration depth of Nb₃Sn) regions of the Nb₃Sn layer. These regions stand in stark contrast to the majority of the thin film grown on the niobium substrate, which is commonly of a thickness between 2-4 μm . Although such features had until now only been seen in cut-outs from the first cavity coated at Cornell [2-4], which showed notably poor performance [5], a more careful analysis of sample coupons coated with Nb₃Sn has shown that these features can also manifest elsewhere, albeit to varying degrees of magnitude.

EXPERIMENTAL METHOD

Detection of the thin film regions is possible using an energy-dispersive X-ray spectroscopy (EDS) map taken in a scanning electron microscope (SEM) while operating at high

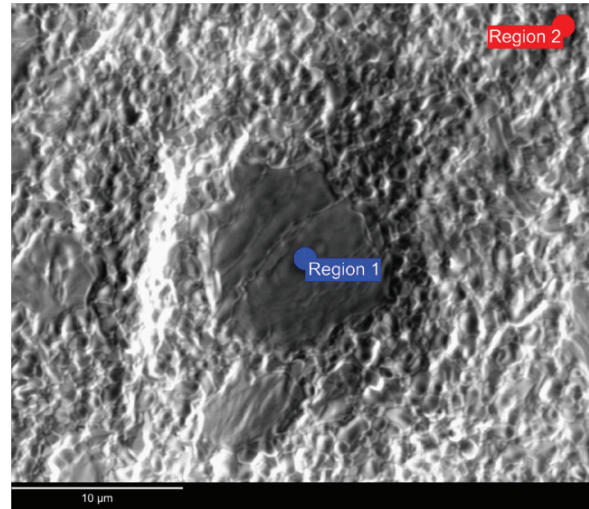


Figure 1: A region of thin Nb₃Sn is seen at the centre of this image taken at 7 kV on the LEO 1550 FESEM. The two region markers indicate the locations of point EDS spectra taken as a function of beam energy.

beam voltage (>20 kV). When operating at high beam voltages, the penetration depth of the beam is greatly increased, to a depth on the order of 1 – 3 μm . For the majority of Nb₃Sn films observed so far, with a thickness of $\approx 3 \mu\text{m}$, this is insufficient to probe the niobium bulk underneath; however, a sufficiently thin film will result in an X-ray spectrum that is possessed of an excess in niobium, as the niobium bulk begins to dominate the X-ray signal.

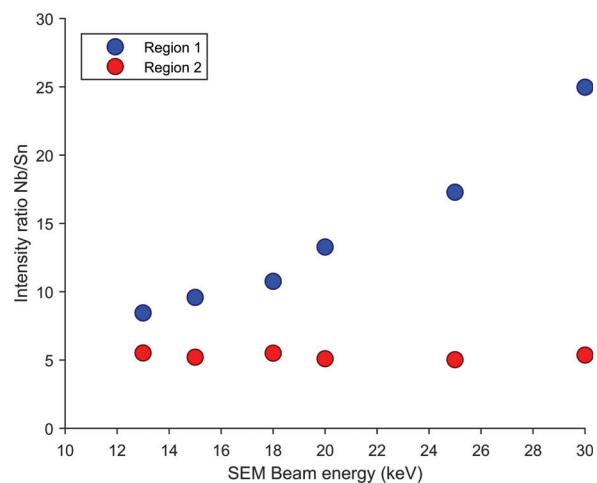


Figure 2: The ratio of the intensity of the niobium to tin signal in the EDS spectra taken in regions 1 and 2 from Fig. 1, as a function of the beam energy.

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This effect is demonstrated in Figs. 1 and 2; upon locating a suspected thin film region in the SEM, as shown in at the centre of Fig. 1, a point EDS spectrum is taken at the centre of the feature at different beam energies. For comparison, an identical point spectrum is taken in a nearby area known to be sufficiently thick and stoichiometric, more representative of the majority of the film surface. As the beam energy is increased, the ratio of the niobium to tin signal measured in region 1 increases. Such a phenomenon is not seen in region 2, indicating that while the Nb_3Sn layer is sufficiently thick in region 2, the feature seen in region 1 is most likely a region where the Nb_3Sn film is significantly less than $1\ \mu\text{m}$. The dependence of the intensity ratio between region 1 and region 2 is shown in Fig. 2.

For the purposes of detecting these thin film regions, a trade-off exists: going to higher beam energies, the signal contrast between normal (i.e. $\approx 3\ \mu\text{m}$) regions of Nb_3Sn and these thin film features increases due to the increased penetration of the beam. Furthermore, increasing beam energy and current increases the count rate, which increases the statistics taken in EDS maps (in which the acquisition time for a single pixel can be dangerously short with regards to the statistics collected) while keeping map acquisition times at reasonable limits. However, as the beam energy and current increase, so too does the lateral probe of the X-ray, which decreases the spatial resolution.

For the images shown in this paper, three SEMs were used, all located at the Cornell Centre for Materials Research (CCMR): an FEI Strata 400 SEM with an integrated FIB column, a LEO 1550 FESEM, and a Tescan Mira3 FESEM. For the purposes of EDS measurements, the first of these is equipped with an Oxford Instruments X-Max 80 mm^2 SDD; the other two are each equipped with a Bruker XFlash 6160 SDD. For EDS mapping, with the intent of identifying regions of thin film, a beam voltage of between 25 and 30 kV was found to be appropriate for mapping regions of between 500×500 to $1000 \times 1000\ \mu\text{m}$ in area. These settings allow the detection of thin film regions with a resolution on the order of $1\text{-}3\ \mu\text{m}$.

THIN FILM REGIONS IN EARLY CAVITY COATINGS

The first cavity coated at Cornell was a single-cell 1.3 GHz single cell cavity of the shape developed for Cornell's Energy Recovery Linac project, designated ERL1-5. The cavity performed comparatively poorly, with an elevated surface resistance and a low quench field [5]. Temperature mapping of the cavity revealed significant surface heating on one of the half-cells, and optical inspection of the half-cell revealed it to be optically shinier than its counterpart. In collaboration with Fermi National Laboratory, coupons were cut out from the cavity, with the locations being decided based upon the temperature maps taken previously [4].

In coupons taken from regions that showed significant heating, EDS maps showed a significant excess of niobium in many regions, as seen in Fig. 3. Although initially thought

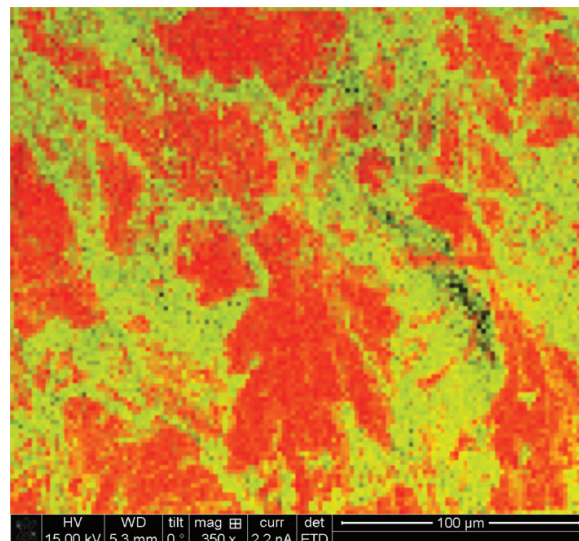


Figure 3: An EDS map taken at 25 kV on the FEI Strata 400 SEM from a sample cut-out taken from ERL1-5. This cut-out was taken from a region that showed substantial heating during RF testing, as seen from temperature mapping [4].

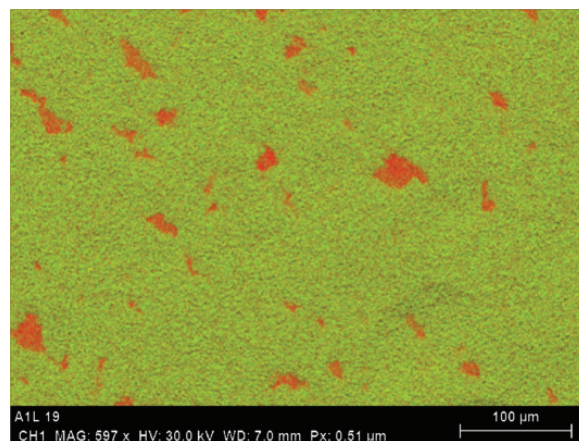


Figure 4: An EDS map taken at 30 kV on the LEO 1550 FESEM of a Nb_3Sn film coated on a large grain niobium substrate.

to be tin-depleted, cross-section analysis showed that instead these regions were significantly thinner than the regions that showed a more stoichiometric ratio of niobium to tin [4]. The conclusion was thus drawn that these “tin-depleted” regions seen in SEM-EDS maps were in fact regions of exceptionally thin film. Image analysis of Fig. 3 reveals that $(65.7 \pm 1.5)\%$ of the area shown is covered in an Nb_3Sn film whose thickness is on the order of the RF penetration depth.

Coupons taken from regions of the cavity that did not show significant heating showed a considerably more uniform coating, with, at the scales observed, no regions of thin film. Since cavities coated after this first attempt showed much improved performance, with no shiny areas, it was assumed that these features were unique – for the time being – to this

cavity. The exact reason that these features formed in certain regions and not others is still the subject of research.

THIN FILM REGIONS IN RECENT SAMPLES

Although originally believed to be relegated to the cavity designated ERL1-5, these thin film regions have recently been detected in other sample coupons coated at Cornell. In particular, a sample of large grain ($>1 \text{ cm}^3$ per grain) niobium coated alongside a fine grain cavity that showed remarkably good performance [6], demonstrated similar features. An EDS map taken from this coupon is shown in Fig. 4, demonstrating a thin film coverage of $(4.0 \pm 0.7)\%$. The thin film features, although not covering a great a percentage of area as seen in Fig. 3, are nonetheless distinctly present and spread over a wide area. In hindsight, however, the appearance of these features on a sample of large grain niobium is not entirely surprising, as some large grain samples coated previously have in cases demonstrated a shininess similar to that seen in the half-cell of ERL1-5.

Separately, a sample of fine grain niobium, coated using the same recipe as utilised for the two most recent cavities, has also shown the presence of these features. However, unlike the other sample coupons presented here, this sample was hung in the furnace without a cavity being present, instead of being placed in the coupon holder located near the tin source. An EDS map of this sample, from which the SEM image seen in Fig. 1 is taken, is shown in Fig. 5, with $(30.0 \pm 2.0)\%$ thin film coverage. A similar fine grain sample, located near the tin source shows much smaller coverage – $< 0.5\%$ – by thin film regions. An EDS map of this sample, associated with a cavity whose performance is given in Ref. [5], is shown in Fig. 6.

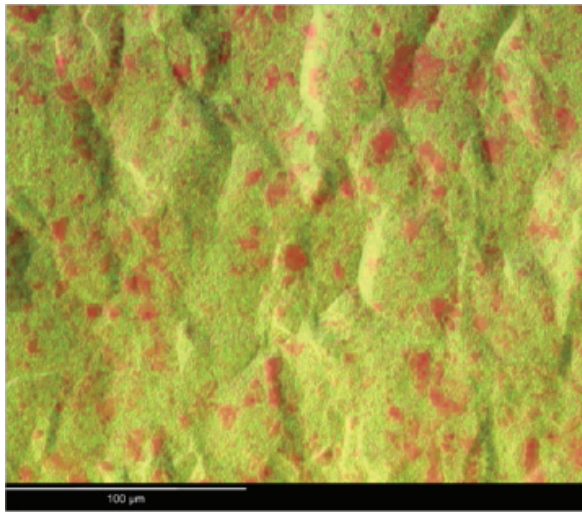


Figure 5: An EDS map taken at 25 kV on the FEI Strata 400 SEM of a Nb_3Sn film coated on a fine grain sample hanging in the middle of the coating furnace.

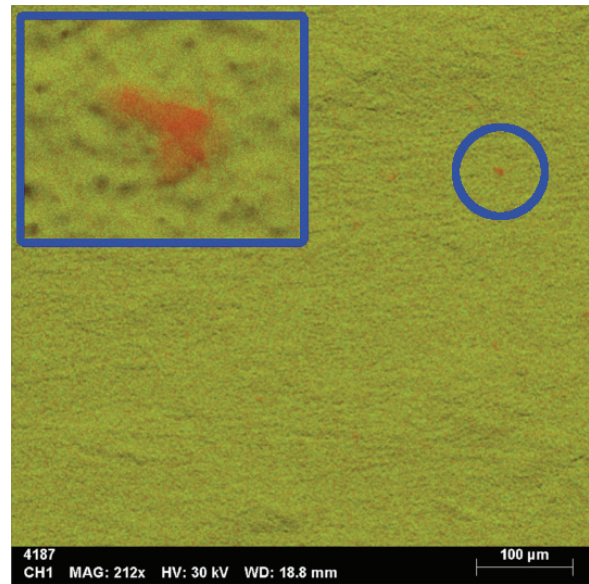


Figure 6: An EDS map taken at 30 kV on the Tescan Mira3 FESEM of a Nb_3Sn film coated on a fine grain sample alongside a cavity that showed good performance. Although the coverage appears to be almost completely uniform, very small regions of thin film do still exist. One such region is shown, inset.

CONCLUSION

The regions of critically thin film seen in the first cavity coated at Cornell have recently been found, to greatly varying degrees of magnitude in terms of surface coverage, in other samples coated with Nb_3Sn . However, at this time the statistics do not exist to be able to draw any conclusions regarding the reason for their appearance, and the degree thereof; only that this phenomenon is no longer unique to the cavity designated ERL1-5. The prevailing suspicion is that the presence of these regions is some function of the underlying substrate; therefore, future studies will now shift to understanding what substrate parameter governs their appearance, and to what degree they harm the RF performance of the resulting cavity.

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