SURFACE ANALYSIS AND MATERIAL PROPERTY STUDIES OF Nb_3Sn ON NIOBIUM FOR USE IN SRF CAVITIES*

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Abstract

Studies of superconducting Nb_3Sn cavities and samples at Cornell University and Argonne National Lab have shown that current state-of-the-art Nb_3Sn cavities are limited by material properties and imperfections. In particular, the presence of regions within the Nb_3Sn layer that are deficient in tin are suspected to be the cause of the lower than expected peak accelerating gradient. In this paper we present results from a material study of the Nb_3Sn layer fabricated using the vapour deposition method, with data collected using AFM, SEM, TEM, EDX, and XRD methods as well as with pulsed RF testing.

INTRODUCTION

Nb_3Sn cavities fabricated at Cornell University have shown good performance without suffering from the Q-slope seen in cavities coated previously [1–3]. In this paper we present results aimed at characterising the material properties of the Nb_3Sn fabricated at Cornell, with an aim to understand current CW quench fields of approx. 65-70 mT seen in the majority of these cavities. Measurements of the surface topology, flux entry field, and phase content are presented here.

FLUX ENTRY FIELD

Measurements of the flux entry field, which at its theoretical limit would be the superheating field H_{sh}, were performed on two separate cavities using Cornell's 1.3 GHz high power pulsed klystron. The 1.3 GHz, single-cell cavities received the same coating recipe, consisting of a 3 hour coating and a 6 hour annealing stage; an in-depth description of the coating apparatus and proces is given in Refs. (CITE TALK AND THESIS). Both showed similar behaviour in vertical tests. A plot of the flux entry field against the square of the bath temperature is shown for the most recently tested cavity is shown in Fig. 1. The results from the previously tested cavity can be found in Ref. [2].

The cavities both show similar behaviour, with two evidently separate linear regions appearing above and below 15-16 K. These have been fitted with the empirically expected approximate temperature dependence of the superheating field,

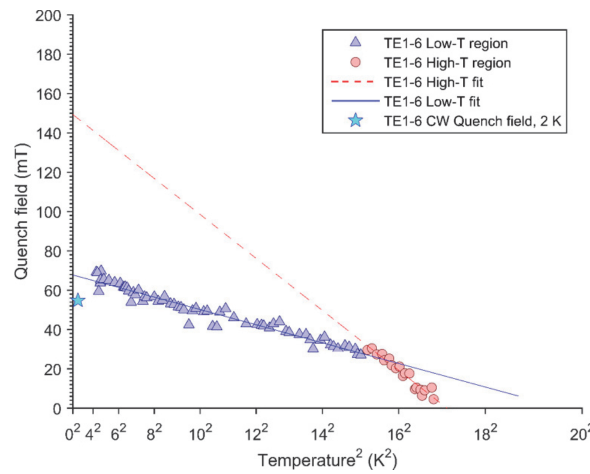

Figure 1: Results from the high pulsed power RF test, showing the flux entry field as a function of bath temperature squared. Both this cavity and that shown in Ref. [2] show two distinct slopes when going from 2^2 to 18^2 K^2.

$$H_{sh}(T) = H_{sh}(0)\left(1 - \left(\frac{T}{T_c}\right)^2\right) . \qquad (1)$$

In both of the high temperature cases, the fit gives a zero-temperature flux entry field, H_{fe}, 150-250 mT, lower than the expected $H_{sh} \approx 400$ mT. Below $15^2 - 16^2$ K^2, the extrapolated H_{fe} is further reduced. This behaviour suggests that two superconductors are in effect, one dominating the RF performance near T_c and the other below $15 - 16$ K.

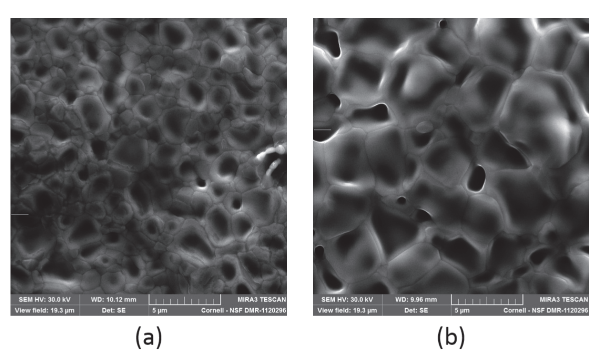

Figure 2: An SEM image of the Nb_3Sn layer for samples from two different coatings which were annealed at different temperatures, a) 1100°C and b) 1200°C.

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It is interesting to note that relative difference in the CW quench fields between the cavity shown in Fig. 1 and that seen in Ref. [2] is less dramatic than the relative difference in the flux entry fields seen in both the high temperature and low temperature regions. This suggests that the quench mechanism may be different between the CW and pulsed measurements.

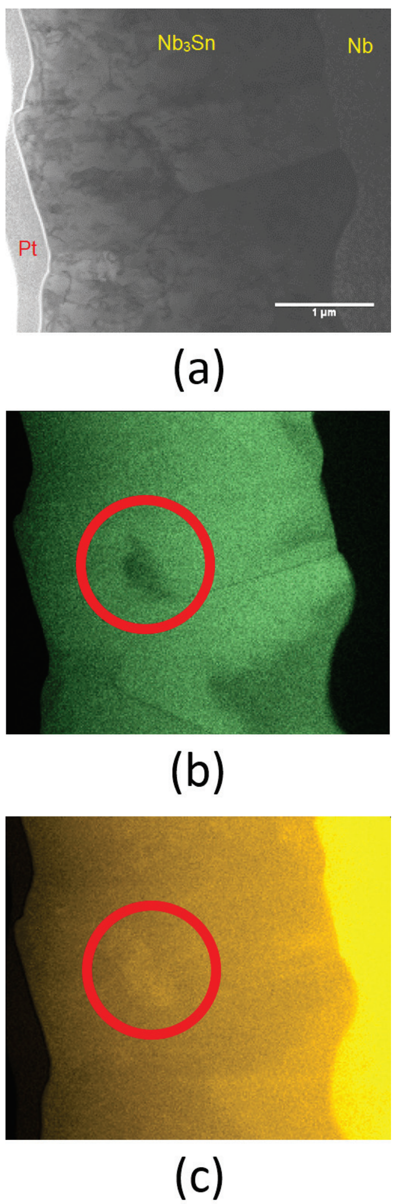

Figure 3: a) A STEM image of a cross-section of a Nb_3Sn sample, with the RF surface (coated with a thin layer of Pt for preparation purposes) on the left and the niobium substrate on the right. An EDS map of b) tin and c) niobium show regions of lower Sn/Nb ratio within the Nb_3Sn layer. Such an area has been highlighted with a red circle.

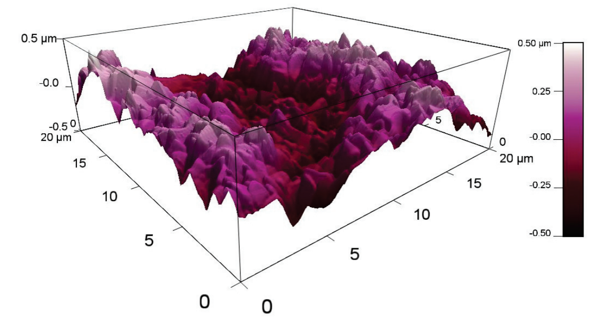

Figure 4: A 3-D AFM image taken of a Nb_3Sn sample, showing a roughness on the order of ± 0.5 μm.

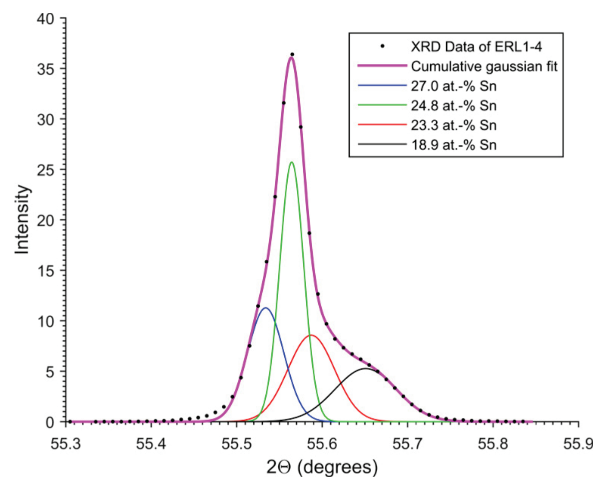

Figure 5: An example of an XRD diffraction peak from a spectrum taken at the APS. The diffraction peak shows evidence of the presence of multiple phases of Nb_3Sn within the layer.

SURFACE TOPOLOGY

The lower than expected flux entry field seen from the high-temperature region fit in Fig. 1 may be explained by the surface topology of the Nb_3Sn layer. Fig. 2 shows a scanning electron microscope (SEM) image of the surface of the layer for two different coatings, which show distinct grains with an average size on the order of $1 - 3$ μm. Surface roughness measurements made using an AFM show considerable roughness, on the order of the grain size seen in the SEM image; a 3-D reconstruction of a surface region is shown in Fig. 4. Further AFM measurements showed no difference in the surface roughness between samples coated on an EP-prepared substrate and a BCP-prepared substrate, suggesting that the roughness is determined by the growth of the Nb_3Sn layer and not the underlying substrate. Surface roughness will result in field enhancement and with it a lower quench field, which could explain the results seen in Fig. 1.

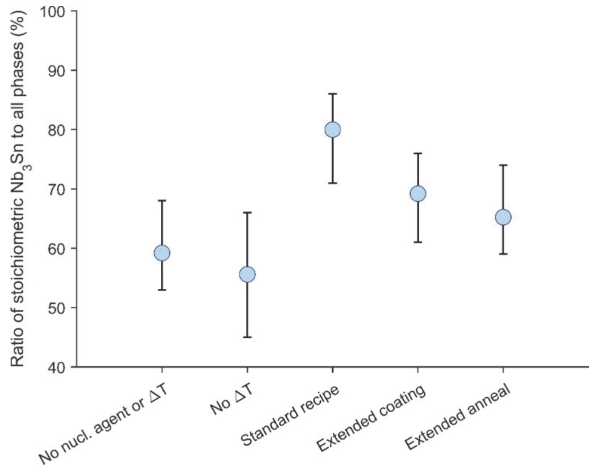

Figure 6: A plot of the ratio (in %) of stoichiometric Nb_3Sn to other phases present within the sample for 5 different coatings. The first two were done with the source being turned on after coating temperatures were reached. In the first, no nucleation agent was used. In the last three, the times were varied for the different stages.

PHASE CONTENT AND TIN-DEPLETED REGIONS

X-ray diffraction (XRD) measurements carried out at Argonne National Lab [4] on samples coated at Cornell have revealed the presence of non-stoichiometric phases of Nb_3Sn within the layer. An example of an XRD diffraction peak showing the presence of these phases is shown in Fig. 5. These measurements have been corroborated by energy-dispersive X-ray spectroscopy (EDS) maps of a transmission electron microscope (TEM) cross-section of the Nb_3Sn layer on the niobium substrate, shown in Fig. 3. EDS maps highlight regions of tin deficiency both within the grains and grain boundaries, with these tin-depleted regions occasionally approaching or breaching the RF surface.

Tin-depleted Nb_3Sn has been seen to posses a significantly lower T_c that its stoichiometric counterpart, even going as low at 6 K [5]. Since Nb_3Sn has a thermal conductivity that is roughly 10^3 lower than that of niobium, these tin depleted regions, locked within this layer but close enough to the RF surface to interact with the RF field, may become thermally unstable even at relatively low fields, leading to the CW quench fields seen around 65-70 mT. These regions, possessed of a lower energy gap and T_c and therefore a lower H_{sh}, may also be responsible for the low-temperature region flux entry field seen in Fig. 1.

Fortunately, it appears that coating parameters can be adjusted to change the ratio of the stoichiometric Nb_3Sn to its tin-depleted counterpart. Shown in Fig. 6 is a plot of this ratio for 5 different coatings. In the first coating, no nucleation agent was used to seed the surface - the coating consisted of a 3 hour coating step in which the source is kept at 1200°C and the cavity is kept at 1100°C, followed by a 6-hour annealing step in which the source is turned off and the entire furnace sits at 1100°C. All other coatings were performed using a nucleation step in which a nucleation agent – $SnCl_2$ – is used to seed the surface prior to ramping to coating temperatures. For the first two coatings, the source was turned on after reaching coating temperatures - for the other 3, a ΔT of 100°C was developed between the source and sample before ramping to coating temperatures. For the last two coatings, in order, the coating step was extended from 3 hours to 6, and the annealing step was increased from 6 hours to 13.

It is evident that the use of the nucleation agent and the presence of a ΔT during the ramp to coating temperatures is necessary to achieve a ratio of > 60%. For the remaining three coatings, initial results suggest that it is both possible to transfer too much tin (in the case of an extended coating), and to over-anneal the sample. Further studies will be undertaken to better understand the dependence of the presence of these phases on the temperatures and times selected for the coating.

CONCLUSION

A study of the material characteristics of the Nb_3Sn films coated at Cornell has revealed the presence of tin-depleted phases of the intermetallic alloy within the Nb_3Sn layer; these phases are known to have a lower T_c than stoichiometric Nb_3Sn. The presence of these phases is felt in pulsed klystron RF tests, which suggest an ultimate flux entry field between 150 and 200 mT. This is significantly lower than the expected flux entry field of Nb_3Sn, although AFM measurements have shown that the surface demonstrates considerable roughness which will lead to field enhancement that lowers the measured flux entry field. The focus of this study will now shift to altering the fabrication parameters of the coating process with the objective of minimising the presence of these bad phases and reducing surface roughness.

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