# $Nb_3Sn$ MATERIALS STUDIES*

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## *Abstract*

$Nb_3Sn$ is a very promising material for use in SRF cavity applications, potentially offering significant improvements in quality factor and energy gradient compared to niobium. In order to better understand how to optimize this material for SRF applications, $Nb_3Sn$ samples were prepared at Cornell via vapor deposition, using varying parameters in the coating process. Microscopic studies were performed with SEM/EDX, and studies were performed on bulk samples to measure secondary electron yield, energy gap, and upper critical magnetic field. The results are presented here, with discussion for how they might point the way towards reaching even higher fields in $Nb_3Sn$ cavities.

## INTRODUCTION

$Nb_3Sn$ is a material with great potential for SRF applications, offering large potential gains in quality factor and energy gradient compared to niobium due to its large critical temperature $T_c$ and predicted superheating field [1]. At Cornell, infrastructure has been developed to coat single cell 1.3 GHz niobium SRF cavities with $Nb_3Sn$ using the vapor deposition technique, as described in [2]. The first cavities produced have had very promising RF performances, achieving quality factors above $10^{10}$ at 4.2 K and 13 MV/m [3]. In order to push performance further, it is important to better understand this material. Its superconducting behavior is determined by its properties on the length scale of the coherence length (approximately 3-4 nm) and the penetration depth (approximately 100 nm), so it must be examined on this scale in order to make correlations to its SRF properties. In this paper, we present the results of both bulk and microscopic studies of $Nb_3Sn$ samples designed to better understand and improve the material and the fabrication process.

## SEM/EDX AFTER NORMAL COATING

The standard coating process (including annealing step discussed in [3]) produces $Nb_3Sn$ grains with size of approximately 2 μm on top of the much larger niobium grains. Figure 1 shows the $Nb_3Sn$ surface imaged by SEM with a tilt of 52 degrees to show the texture. There are flat facets visible in many grains, as well as smooth curves, and many of the grains have what appear to be indented regions near the middle. EDX scans performed in several regions show a stoichiometry of 24±1 atomic percent tin.

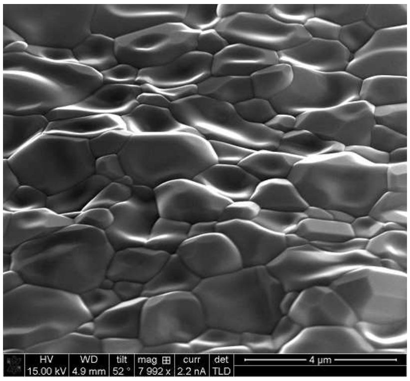

Figure 1: $Nb_3Sn$ surface after coating with the standard recipe. The sample is tilted at 52 degrees with respect to the detector to highlight three dimensional features.

## SEM/EDX WITH LG SUBSTRATES

Several small samples were fabricated for experimenting with different $Nb_3Sn$ coating conditions, including some made from large grain niobium. After coating, these samples showed some structures not observed on fine grain niobium. SEM images show the 1-2 μm sized grains usually observed along with localized "clumps." These regions, which appear as dark areas with size on the order of 10 μm, have a lower tin content, as measured by EDX, by up to 10 atomic percent. Two images taken from samples coated without the usual nucleation agent, $SnCl_2$, are shown in Fig. 2 (these structures are also observed on LG samples when coating with $SnCl_2$).

In RF measurements of cavities, a dramatic reduction in $Q$-slope was observed after an annealing step was added, indicating that the grain structure may strongly influence RF properties. Therefore, studies like this are important to understand how the substrate and coating process influence the grain structure.

## SEM/EDX BEFORE/AFTER HF RINSE

For the first time, a niobium cavity with an electropolished surface was coated with $Nb_3Sn$, and EP samples were coated with it. An SEM image is seen in Fig. 3. The sample was later given 5 cycles of HF rinsing, the same treatment given to a cavity that caused it to have strong $Q$-slope. SEM of the HF-rinsed surface showed unusual structures on top of the $Nb_3Sn$ grains, which might be the source of the extra losses. EDX was used to probe the structures, but no sign of contamination was measured, and no change in atomic

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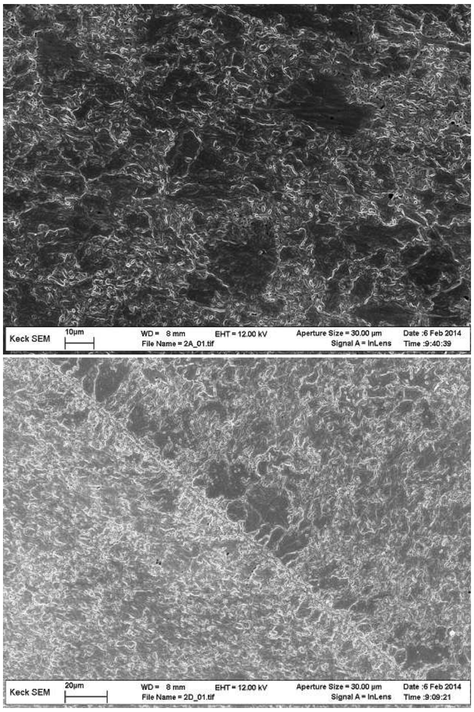

Figure 2: Large grain niobium coated with $Nb_3Sn$ appears to have some regions with large "clumps" along with the usual grains. Shown is a sample coated without nucleation agent, both far from (top) and including (bottom) a grain boundary of the LG substrate. The niobium grain boundary's influence on the grain structure of the $Nb_3Sn$ is clearly observed.

percent composition of the elements normally observed on the surface (Nb, Sn, O, and a small amount of C) was observed within the resolution of the tool. Auger analysis is planned to achieve higher resolution. It is hoped that these studies will lead to a better understanding of the cavity performance degradation so that a defect removal process can be developed that does not cause $Q$-slope.

## TEM SAMPLE PREPARATION

TEM with elemental analysis would allow detailed studies to be done of very small features, such as grain boundary structure and composition, change in tin concentration as a function of depth, and the interface of niobium with $Nb_3Sn$. To be able to transmit electrons through a sample, it must be very thin, on the order of tens of nm, making TEM samples very difficult to prepare. Two methods were attempted, shown in Fig. 4: mechanical polishing with a diamond lapping film and ion milling via focused ion beam (FIB). The mechanically polished sample was TEM-transparent, but EDX revealed no remaining tin. It is possible that the $Nb_3Sn$ layer was removed during polishing. The first sample prepared with FIB was too thick to allow transmission. Next steps will focus on preparing a thin enough sample with FIB.

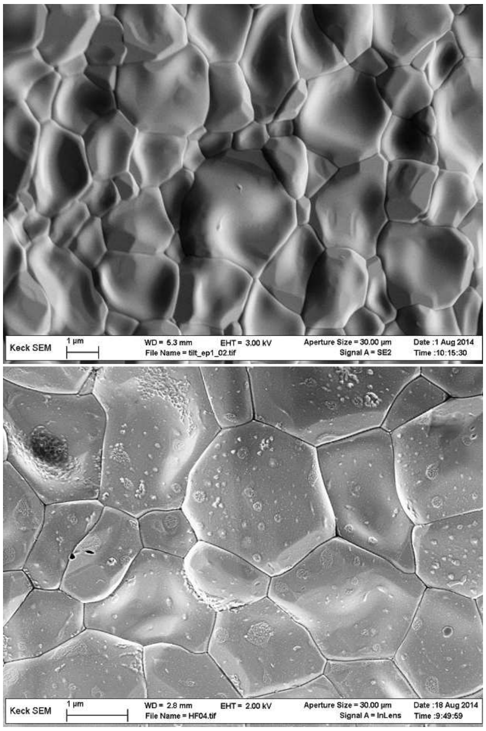

Figure 3: $Nb_3Sn$ coated on an EP Nb surface before (top) and after (bottom) HF rinsing. Note the structures found after rinsing.

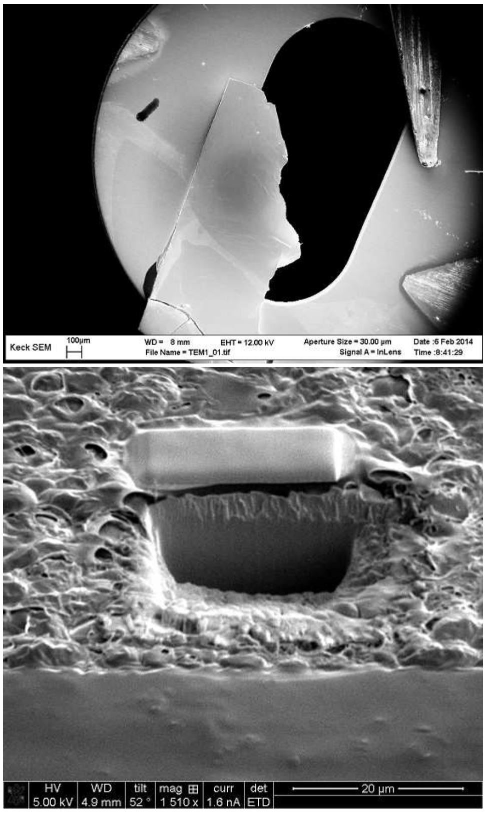

Figure 4: Attempts to make TEM-transparent samples via mechanical polishing with a diamond lapping film (top) and ion milling (bottom). Further effort will be needed to extract usable TEM samples.

## BULK MEASUREMENTS

Secondary electron yield (SEY) measurements were performed by S. Aull at CERN on a $Nb_3Sn$ sample provided by Cornell. The maximum SEY was found to be 2.5, very similar to niobium, suggesting that multipacting in $Nb_3Sn$ cavities should be comparable to that normally observed in Nb cavities [4].

Point contact tunneling (PCT) measurements were performed at Argonne on a $Nb_3Sn$ sample provided by Cornell. Correlations with temperatures gave an energy gap $\Delta$ at zero temperature of approximately 2.89 meV with a $T_c$ of 16 K. These values (combined with composition from EDX) appear to follow correlations found in the literature [5], as shown in Fig. 5. Note that the PCT curves show a nice, sharp gap at low temperatures.

$H_{c2}$ measurements were also performed at Argonne on the same Cornell $Nb_3Sn$ sample, using a 50% resistive criterion (where the transition is taken to occur when the resistance is half of the normal conducting value). As shown in Fig. 6, the extrapolated value for $H_{c2}$ at zero temperature of 17.9 T appears to follow correlations found in the literature [5]. $H_{c2}$ calculated from material parameters extracted from impedance measurements during RF tests is also shown [3].

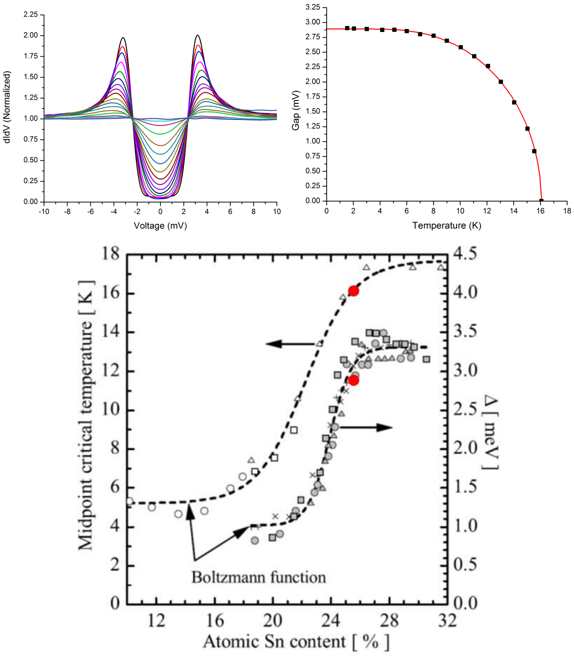

Figure 5: Top left: Point contact tunneling measurements made at several different temperatures. Top right: extraction of $T_c$ and $\Delta$. Bottom: Comparison of extracted values (red) to literature with figure adapted from [5].

## CONCLUSIONS

SEM/EDX studies were performed on $Nb_3Sn$ coatings on large grain substrates and electropolished substrates. The large grain substrates showed broad “clumps” of low tin content material. SEM of HF rinsed samples showed unusual structures, perhaps explaining performance degradation observed in RF tests after HF rinse. SEY, PCT, and high magnetic field measurements were performed on bulk samples. SEY measurements indicate that vulnerability to multipacting should be similar to that in niobium cavities. $\Delta$ and $H_{c2}$ extracted from PCT and magnetic measurements appeared to follow trends from the literature, demonstrating the high quality of the $Nb_3Sn$ coatings fabricated at Cornell.

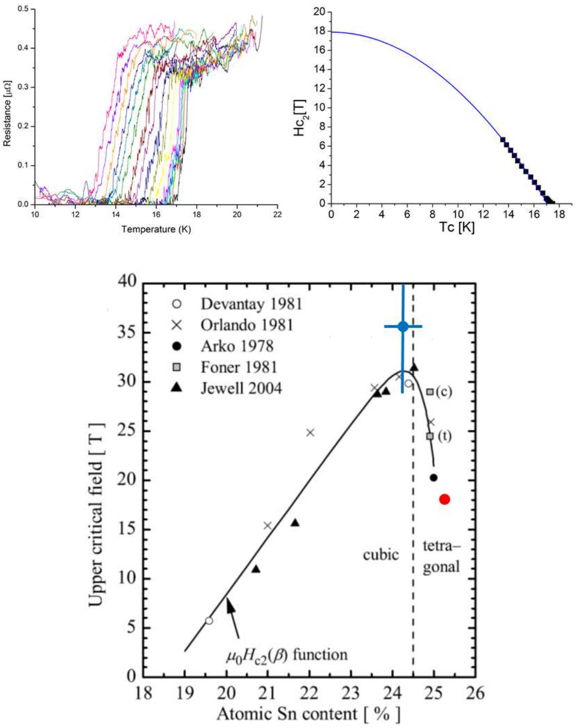

Figure 6: Top left: Resistive measurements made at several different external fields. Top right: extraction of $H_{c2}$ extrapolated to zero temperature with 50% resistive criterion. Bottom: Extracted value from $H_{c2}$ measurements (red) and computed value from material parameters generated extracted from impedance measurements during RF tests (blue) are compared to literature with figure adapted from [5].

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