

CORNELL SRF NEW MATERIALS PROGRAM *

S. Posen[†], M. Liepe, and Y. Xie, CLASSE, Ithaca, NY, USA

Abstract

The SRF group at Cornell has recently pioneered an extensive program to investigate alternative materials for superconducting cavities. We have developed facilities to fabricate Nb₃Sn, a superconductor which will theoretically be able to reach more than twice the maximum accelerating field of Nb in a cavity under the same operating conditions. In addition, with the critical temperature of Nb₃Sn being twice that of Nb, Nb₃Sn would allow operation of SRF cavities with a much higher cryogenic efficiency. We have also manufactured two TE cavities that measure the RF properties of small, flat samples, ideal for material fabrication methods in development. This paper presents an overview of the materials research program. First results from tests of Nb₃Sn samples are presented.

INTRODUCTION

After years of development, niobium SRF cavities are now being fabricated that are limited in gradient by the RF critical field, not multipacting, thermal instability, field emission, or high field Q slope. To reach even higher gradients, new materials must be used that will allow the RF surface to tolerate higher magnetic without quench. Theory [1] predicts that coating a cavity with Nb₃Sn will allow it to reach twice the accelerating gradient of a niobium cavity. Nb₃Sn also benefits from a critical temperature that is nearly twice as high as that of niobium, which allows cavities coated with Nb₃Sn to operate with quality factors far exceeding those of Nb cavities at the same temperature.

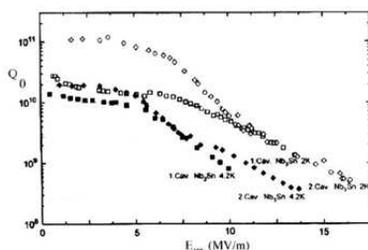


Figure 1: The best results obtained at University of Wuppertal for 1.5 GHz cavities vapor-diffusion coated with Nb₃Sn. From [3].

The great potential of Nb₃Sn-coated cavities has already been shown in the results obtained by researchers at University of Wuppertal in the 1980s-90s [2]. They produced cavities with quality factors of 10¹¹ at 2 K and accelerating

gradients competitive with the gradients of niobium cavities produced at the time. Some vertical test results are shown in Figure 1. Guided by their work, facilities at Cornell have been developed to fabricate Nb₃Sn.

COATING PROCESS

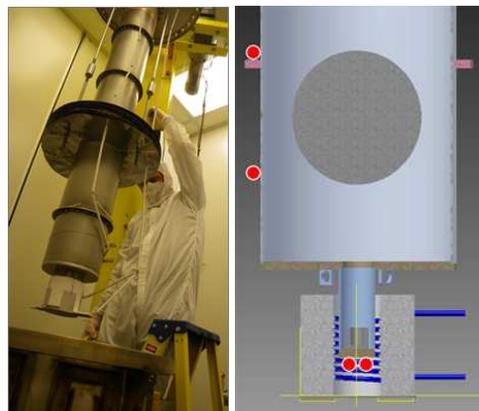


Figure 2: The Nb₃Sn coating chamber being inserted into the UHV furnace (left) and a cross section (right) showing the tin crucible surrounded by the heater and the substrate suspended above. The red circles represent thermocouples for temperature monitoring.

The vapor diffusion coating process was based on [2]. A UHV furnace containing a niobium substrate, a tungsten crucible containing 99.999% Sn pellets, and a tungsten crucible containing a small amount of SnCl₂ is evacuated and degassed at 200°C. The coating chamber, shown in Figure 2, is self-contained to avoid contaminating the furnace with tin. The valves to the pumps are then closed and the temperature is raised to 500°C, at which point the vapor pressure of SnCl₂ is high enough for it to create tin nucleation sites on the Nb surface. After 5 hours, the furnace temperature is raised to 1100°C, and a heater raises the temperature of the Sn slightly higher, to 1200°C. The elevated temperature from the heater increases the vapor pressure of tin in the coating chamber. The furnace temperature and the tin temperature control the rate of interdiffusion of niobium and tin in the sample and the rate at which tin arrives at the sample, and they can be varied to affect the composition of the alloy produced. After 3 hours the heater is turned off, and the remaining tin vapor in the chamber is given 30 minutes to reach a surface and diffuse in, so as to avoid unreacted tin on the RF surface. Finally, the furnace is turned off and backfilled with nitrogen gas when it is cool.

The Cornell procedure will include preparation techniques that have been developed in the last two decades. Already, Nb substrates—currently only flat samples have

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[†] sep93@cornell.edu

been coated, not full cavities—are HPR'd prior to coating. Soon, substrates will be EP'd to give a smooth RF surface prior to coating, and also baked at 120°C.

ANALYSIS OF Nb₃Sn COATINGS

One of the samples was anodized to test the surface composition. In this procedure, the sample is placed in 10% NH₄OH and a 75 V potential is applied between it and an anode. The thickness of the oxide that grows gives it a color that is characteristic of the material on the surface. Any unreacted tin will turn yellow, niobium will turn blue, and Nb₃Sn will turn pink-purple [4]. The color of the sample, shown in Figure 3, indicates that the material on the surface is Nb₃Sn.

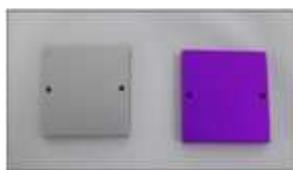


Figure 3: Nb₃Sn-coated sample (left) and an identical sample after anodization in NH₄OH at 75 V (right). The pink-purple color indicates Nb₃Sn phase on surface.

SEM images of the first samples coated are shown in Figure 4. Micron-sized grain growth was observed similar to that in images published by the group at Wuppertal.

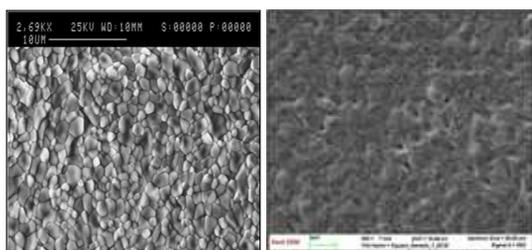


Figure 4: SEM images of Nb₃Sn produced at Wuppertal in 1996 (left) and at Cornell in 2011 (right).

To quantify the composition of the Nb₃Sn layer, energy dispersive x-ray (EDX) analysis was performed. Firing 10 keV electrons at the sample and measuring the spectrum of x-rays emitted (shown in Figure 5), it probes the relative abundance of Nb and Sn up to approximately 0.5 microns into the surface [5]. An abundance of 24.2±0.5 atomic percent tin (atm%Sn) was measured (uncertainty from [6]).

Nb₃Sn is the name given to the A15 phase of the Nb-Sn system, which has a composition ranging from approximately 18 to 25 atm%Sn. A. Godeke has reviewed the strong variation in the properties of the alloy with composition [7]. For example, the variation of T_c and H_{c2} with composition is shown in Figure 6. The data indicate that higher Sn content will yield a higher T_c , which is desirable to reduce the BCS resistance. Godeke notes, however,

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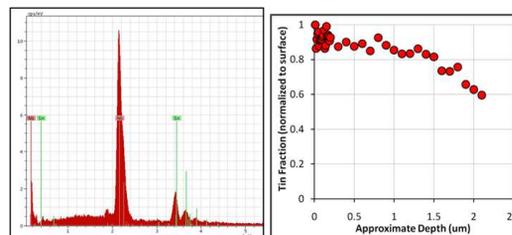


Figure 5: EDX spectrum of Nb₃Sn sample indicates composition ~24.2 atm%Sn (left); alternately sputtering and performing XPS scans shows atm%Sn versus depth (right).

that above 24.5 atm%Sn, the alloy will undergo a spontaneous lattice distortion at low temperatures which can lead to strain and, Godeke suggests, reduced RF performance [8]. Godeke recommends alloying close to 24.5 atm%Sn but below it, which means that the coating produced by the Cornell furnace is right in the desired composition range.

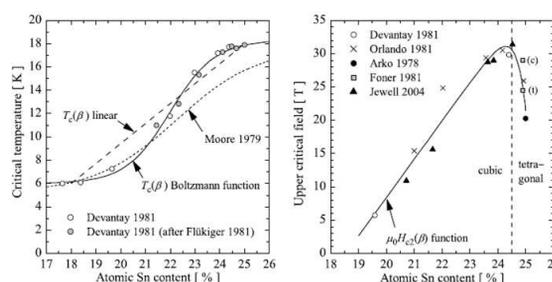


Figure 6: Literature data for T_c versus composition (left) and H_{c2} versus composition (right). From [8].

By alternatively performing X-ray photoelectron spectroscopy (XPS) scans and sputtering the sample with Ar⁺ ions, the variation in composition with depth could be studied. The results in Figure 5 show that the Nb₃Sn layer has approximately uniform composition up to a depth of ~1.5 μm. The first surface scan is not shown as it showed foreign elements, likely from handling outside the clean room. The composition is normalized to the scan after the first etch, as differential sputtering rates give the absolute composition very large uncertainty.

To better characterize the Nb₃Sn layer, the critical temperature of one of the samples was determined using a 4-wire measurement. The plot of resistance versus temperature below 26 K is shown on the left in Figure 7. A large discontinuity in resistance indicating a superconducting transition occurs at 18.1±0.2 K. This is very close to the highest recorded value for Nb₃Sn, 18.3 K [7]. No discontinuity is observed at any other temperature, indicating that a complete path is available between the leads with this critical temperature. The non-zero resistance below the transition at 18.1 K may result from not having the correct reference phase on the lock-in amplifier used in this experiment to remove any reactive components of the impedance.

In the same 4-wire measurement, an estimate was made of the RRR degradation in the Nb substrate resulting from

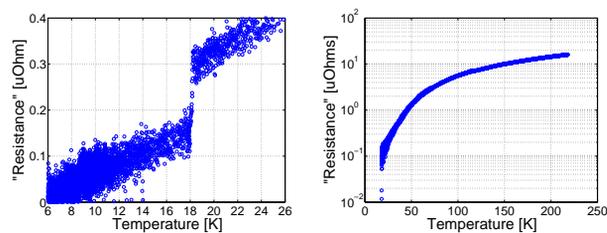


Figure 7: 4-wire measurement of the Nb₃Sn sample indicates a critical temperature of ~ 18.1 K (left); resistance of sample versus temperature shows that minimal RRR degradation occurred during coating process (right).

the coating process. During the coating process, the valves to the pumps are closed when the furnace temperature is above 200°C, potentially allowing residual gases to reduce the RRR of the substrate, which was initially 280. To check for this, the resistance of the sample was monitored from the transition up to room temperature, as plotted in the right of Figure 7. The normal resistance at 4.2 K could not be measured, but a lower bound for the RRR of 210 could be obtained using the resistance just above the transition. This shows that the RRR degradation during coating is minimal.

RF TESTING

Very important to guiding the development of alternative materials is RF testing of small samples with a simple geometry. These tests will provide a method of correlating performance to surface analysis results and systematically improve the coating process without having to build a new cavity each time.

Two sample testing cavities have been built at Cornell, shown in Figure 8. The first is a 6 GHz niobium TE pillbox cavity with a 3.75" diameter sample plate incorporated into it as the bottom wall. During commissioning tests, it reached fields higher than 30 mT on the sample, a world record for this type of cavity. The second cavity is a TE mushroom cavity with a 5" diameter sample plate that operates in two modes, at approximately 5 and 6 GHz. The two modes produce maximum surface magnetic fields 1.24 and 1.57 times higher on the sample than on the niobium. The cavity will undergo first tests next month. T-mapping apparatuses have been developed for the cavities to provide precise measurement of surface resistance. See [9] for details.

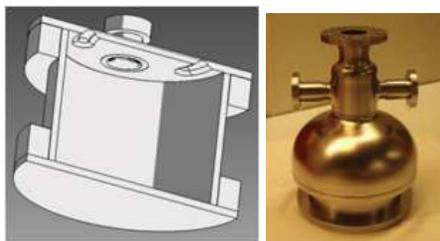


Figure 8: CAD section view of the pillbox cavity (left) and picture of the mushroom cavity (right).

A Nb bottom plate for the pillbox cavity was coated with Nb₃Sn so that RF tests could be performed. Images before and after coating are shown in Figure 9. It received a 130 μm BCP and HPR before coating. A small witness sample was coated with the bottom plate, which showed a composition of 23.6 ± 0.5 atm%Sn under EDX analysis—though its tin content is likely somewhat smaller than that of the bottom plate, which was closer to the tin source—and turned pink-purple when anodized. These tests demonstrate the reliability of the coating process to produce Nb₃Sn of the desired composition. RF testing will begin as soon as commissioning of the pillbox cavity is complete.

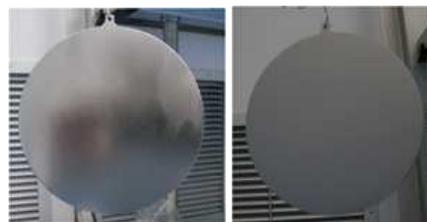


Figure 9: Pictures of pillbox cavity bottom plate before (left) and after (right) Nb₃Sn coating.

CONCLUSIONS

Very promising results have been obtained on the first samples produced by the Nb₃Sn program at Cornell. T_c measurements, EDX, and anodization all suggest that the desired composition has been achieved. The next step will be RF testing in Cornell's TE pillbox and mushroom cavities. Fabrication of a full single-cell cavity is planned for 2012. Full-cavity T-mapping will be employed during testing to identify spots with reduced RF performance, and any such spots will be cut out of the cavity to identify coating defects and find a way to remedy them.

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