

AN INNOVATIVE Nb₃Sn FILM APPROACH AND ITS POTENTIAL FOR SRF APPLICATIONS *

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Abstract

A novel electro-chemical technique to produce Nb₃Sn films on Nb substrates was developed and optimized at Fermilab. The Nb₃Sn phase is obtained in a two-electrode cell, by electrodeposition from aqueous solutions of Sn layers and Cu intermediate layers onto Nb substrates. Subsequent thermal treatments in inert atmosphere are realized at a maximum temperature of 700°C to obtain the Nb₃Sn superconducting phase. Several superconducting Nb₃Sn films were obtained on Nb substrates by studying and optimizing most parameters of the electro-plating process. Samples were characterized at Fermilab, NIMS, KEK and JLab, including SEM/EPMA analyses, DC and inductive tests of critical temperature T_{c0} , and lower critical field H_{c1} (4.2 K) by SQUID. In parallel to sample development and fabrication at FNAL, at JLab and KEK effort was put into etching and electro-polishing techniques adequate to remove the Cu and bronze phases from the samples' outer surface. This is necessary prior to measurements at JLab of the surface impedance of flat samples in a setup that make use of an RF host cavity.

INTRODUCTION

The two most important aspects of SRF performance are the accelerating field E_{acc} and the cavity quality factor, Q_0 . The accelerating gradient in SRF cavities is proportional to the peak magnetic field on the cavity wall. At RF frequencies the peak magnetic field is limited by the metastable superheating field, H_{sh} [1]. The maximum accelerating gradient expected for Nb cavities is ~ 40 MV/m. Nb₃Sn is an attractive alternative because it has a high T_{c0} of up to 18 K and in a binary form an H_{c20} of 23T or larger. With a theoretical H_{sh} expected to be double that of Nb, SRF cavities with a thin layer of Nb₃Sn coated onto their inner surface should produce larger accelerating gradients than Nb. Fig. 1 shows a theoretical comparison of Q_0 for 1.3 GHz Nb₃Sn and Nb cavities, which illustrates the effect of the larger T_{c0} of Nb₃Sn on Q_0 . The larger T_{c0} of Nb₃Sn has also the advantage of allowing the cavities to operate at 4.5 K rather than at ~ 2 K that is used for Nb cavities to obtain a higher gradient. This means less expensive refrigeration and more cryogenic reliability.

Nb₃Sn coated SRF cavities have been demonstrated to improve the cavity quality factor, but have not produced the theoretically expected gradient. Perhaps the longest

standing Nb₃Sn technique for SRF is that of a vapor diffusion process followed by thermal reaction at high temperature to form the Nb₃Sn [2, 3]. It consists of five stages, including degassing at 100° to 200°C to remove moisture; nucleation at intermediate temperature; ramp up to and holding at a coating temperature between 1100°C and 1300°C for Nb₃Sn formation; and finally annealing to diffuse excess tin. This process has so far produced single cell and double cell cavities only because the scale-up to full assemblies is particularly challenging.

In this paper an alternate process, that of chemical electrodeposition, followed by reaction in inert atmosphere, will be described. The electroplating is performed at near room temperature and at atmospheric pressure. The advantage of electrodeposition is its simplicity, accurate control, and low costs. Also, it can be performed on any 3D surface such as the inner surface of SRF cavities. Electrodeposition should be among the least expensive ways to produce SRF cavities. It should also provide a more uniform Nb₃Sn coating. Improving the quality of the Nb₃Sn could reduce the large gap between the theoretical and measured H_{sh} .

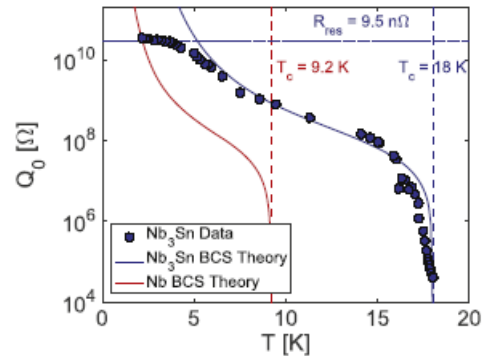


Fig. 1: Calculation of Q_0 for 1.3 GHz SRF cavities with Nb₃Sn and Nb inner surfaces [4].

EXPERIMENTAL METHOD

An electro-chemical deposition technique, which had been collaboratively developed with Politecnico di Milano [5, 6] and is under patenting process, to produce superconducting Nb₃Sn films on Nb substrates, was reproduced at Fermilab. In the past year, the equipment and consumables were procured, and the complete electro-chemical system was commissioned.

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In electroplating the metallic coating is deposited on another metal surface through an electrolyte solution. The metal to be plated acts as the cathode and when the appropriate current is applied, positively charged ions traveling from the anode into the solution will discharge and get deposited on the cathode until a film of desired thickness is formed. Fig. 2, top shows a schematic with two symmetric anodes for bilateral coating of flat samples. Fig. 2, bottom shows the concept for scale-up to 3D surfaces, such as internal coating of cylinders, as first step to the internal coating of SRF cavities.

Fig. 3 shows the sequence of deposited layers. First, using the Nb as cathode and Cu anode, a thin seed layer of Cu is deposited in an acid solution. The Cu lowers the formation temperature for the A15 compound and suppresses the unwanted NbSn₂ and Nb₆Sn₅ phases. In a second electroplating step, the resulting Nb/Cu sample is used as cathode, and a thick layer of Sn is deposited with a Sn anode within a commercial Sn-rich solution. And finally, on the resulting Nb/Cu/Sn sample, a Cu layer is again deposited using a Cu anode. Each electrodeposition step is carried out at near room temperature and at atmospheric pressure. Details are illustrated in [6].

Nb₃Sn is formed through solid diffusion by reacting the multi-layered samples in inert atmosphere (argon) using the temperature profile of Fig. 4, at a maximum temperature of 700°C.

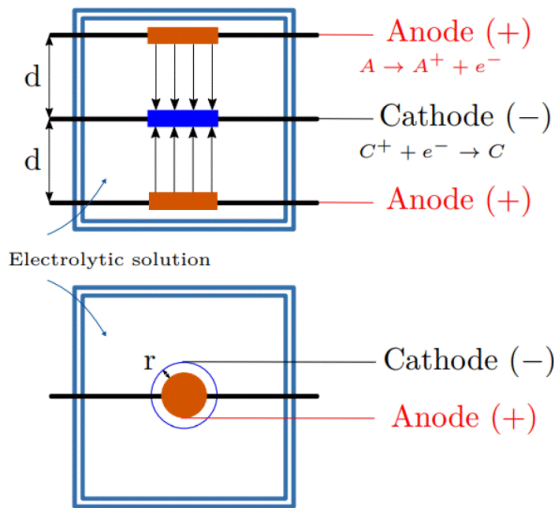


Fig. 2: Schematic of electro-chemical cell for bilateral coating of Nb flat samples (top), and internal coating of Nb cylinders (bottom).

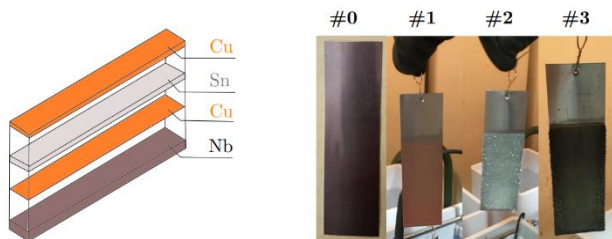


Fig. 3: Sequence of deposited layers (left), and pictures of sample at each deposition step (right).

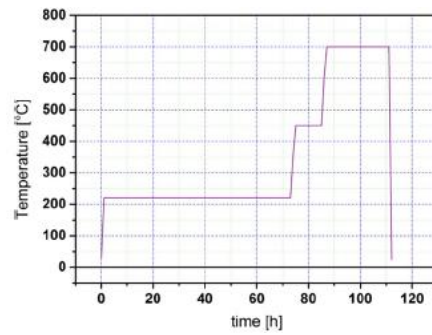


Fig. 4: Temperature profile during Nb/Cu/Sn/Cu solid diffusion.

Reacted Nb₃Sn samples were characterized using a Scanning Electron Microscope (SEM) and Electron Probe Microanalysis (EPMA), transport test of critical current $I_c(B)$ up to 14 T to determine the upper critical field B_{c20} as a free parameter in the $I_c(B)$ data fitting, resistive and inductive critical temperature T_{c0} , as well as SQUID measurements of $H_{c1}(4.2K)$.

Reacted Nb₃Sn film samples were also sent to JLab and to KEK for developing the appropriate etching and/or electropolishing techniques to remove the few micrometers of Cu and bronze phases at the samples' outer surface.

RESULTS

Several superconducting Nb₃Sn films of thickness of up to 12 μm were obtained on Nb substrates of 0.3 mm to 0.5 mm thickness by studying and optimizing most parameters of the electro-plating process, including:

- Bath composition and anode materials for each of the three deposition steps;
- Current densities, deposition times, stirring rates, and cathode and anode relative orientation in DC mode;
- Current densities, deposition times, stirring rates, cathode and anode relative orientation, pulse frequencies, and duty cycles in pulsed mode.

The pulsed mode was eventually chosen for all three deposition steps.

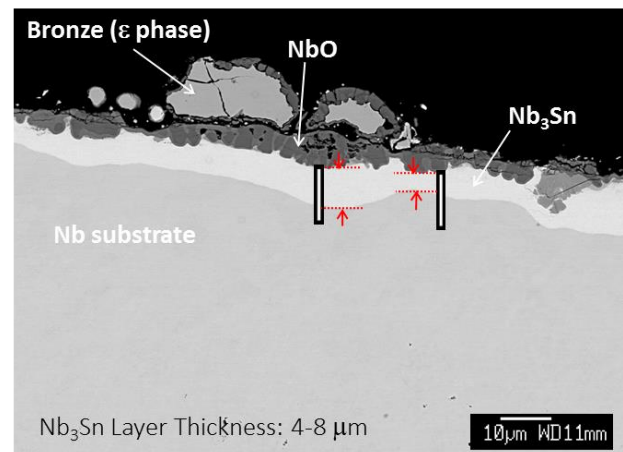


Fig. 6: SEM cross-section of a sample (NIMS).

Fig. 6 shows an SEM picture of the longitudinal cross section of a sample (sample No. 23). EPMA was also performed and allowed identification of bronze phases as well as Nb oxides on the outer surface of the Nb₃Sn layer. Clearly, for SRF operation these residues must be removed by either etching, electropolishing or mechanically. Fig. 7 shows the T_{c0} result of 17.5 K (defined as in [6]) obtained for the same sample as measured resistively. The same sample was also measured by SQUID (Fig. 8) and the resulting T_{c0} was 17.6 K. The two results are remarkably consistent. Fig. 9 shows the $M-H$ curve obtained with the SQUID magnetometer. The $H_{c1}(4.2K)$ for this sample was 600 \AA .

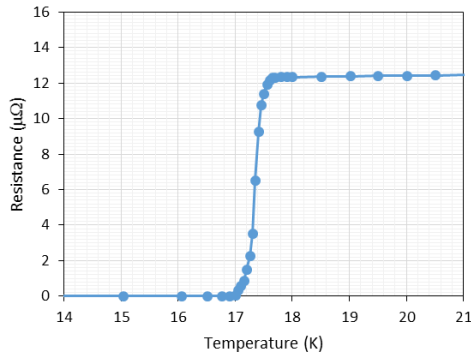


Fig. 7: DC test at FNAL of $T_{c0} = 17.5$ K of a Nb₃Sn film sample.

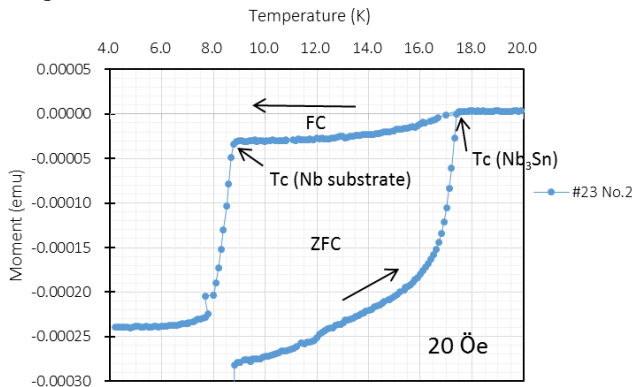


Fig. 8: $M-T$ curve obtained with SQUID magnetometer at NIMS. The $T_{c0} = 17.6$ K.

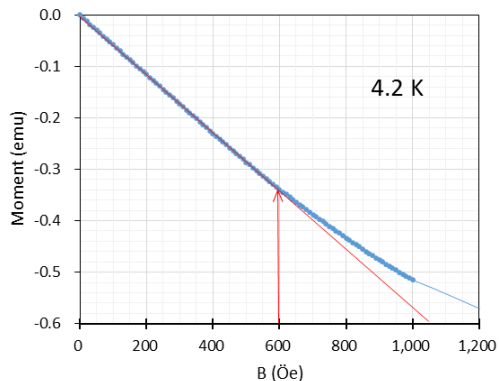


Fig. 9: $M-H$ curve obtained with SQUID magnetometer at NIMS. The $H_{c1}(4.2\text{ K}) = 600$ \AA .

For this sample, an upper critical magnetic field, B_{c20} , of 23.2 T was obtained as a free parameter in the $I_c(B)$ data fitting. The measured T_{c0} value of 17.5 K was used as a fixed parameter in the fit.

It is important to know how well these thin film samples would perform in a cavity. A TE011 cavity with a demountable endplate, where a disc sample can be placed, can be used for the measurement of the surface impedance. A cavity test facility of this type exists at JLab [7].

CONCLUSIONS

High gradient superconducting cavities (SRF) will be needed for future accelerators. However, the accelerating field of Nb cavities is limited by the peak magnetic field on the cavity surface. Nb₃Sn SRF cavities should produce larger accelerating gradients and a larger quality factor, Q_0 , than Nb cavities. Also, the higher T_{c0} of Nb₃Sn allows cavities to operate at 4.5 K rather than ~ 2 K that is used for Nb cavities to obtain a higher gradient. This means less expensive refrigeration and more cryogenic reliability.

Nb₃Sn coated SRF cavities, with Nb₃Sn produced by vapor diffusion followed by a thermal reaction at high temperature, have achieved about half the theoretical predicted gradient. Nb₃Sn plated cavity performance may be improved by using electrochemical deposition. The advantages are its simplicity, accurate control, and low costs. Electrodeposition should be among the least expensive ways to produce SRF cavities, and it should provide a more uniform Nb₃Sn coating. Improving the quality of the Nb₃Sn could reduce the large gap between the theoretical and measured H_{sh} . Electrochemical techniques may also eliminate the problem of a low Sn concentration at the Nb interface, which is a fundamental limit of the current vapor deposition techniques. Lastly, electrodeposition can be performed on any 3D surface such as the inner surface of SRF cavities. Nb₃Sn coating experiments of Nb cylinders are under way.

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