DEVELOPMENT OF HIGH-PERFORMANCE NIOBIUM-3 TIN CAVITIES AT CORNELL UNIVERSITY

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Abstract

Niobium-3 tin is a promising material for next-generation superconducting RF cavities due to its high critical temperature and high theoretical field limit. There is currently significant worldwide effort aiming to improve Nb_3Sn growth to push this material to its ultimate performance limits. This paper will present an overview of Nb_3Sn cavity development at Cornell University. One approach we are pursuing is to further advance the vapor diffusion process through reducing film thickness and optimizing the nucleation step to grow more uniform layers of stoichiometric Nb_3Sn. Additionally, we are exploring alternative growth methods, such as the development of a plasma-enhanced chemical vapor deposition (CVD) system, as well as Nb_3Sn growth via electrochemical synthesis.

INTRODUCTION

Niobium-3 tin (Nb_3Sn) has the potential to push the performance of next-generation SRF cavities beyond that of niobium due to its remarkable superconducting properties [1]. With twice the critical temperature of traditional Nb used in present-day SRF accelerator cavities, Nb_3Sn opens the possibility of higher operating temperatures, significantly reducing the cooling requirements and consequently having greater operational efficiency. Furthermore, this material can in theory withstand almost twice the RF fields than the typical Nb SRF cavity, hence doubling the maximum accelerating gradient achievable and allowing for shorter and more powerful accelerators [1, 2].

However, achieving optimal growth of this material remains an immensely challenging task. In fact, the development of Nb_3Sn SRF cavities has been a topic of significant research and progress since the 1970s. Early attempts by institutions like Siemens AG, Karlsruhe, and Wuppertal faced challenges such as the presence of a strong Q-slope at higher fields. Nevertheless, after a pause in research, Cornell University took the lead in resuming Nb_3Sn cavity development in 2009, through implementing an adapted Wuppertal growth method of this material [3, 4]. Cornell's efforts successfully tackled the Q-slope issue and showcased the material's potential for pushing the performance of SRF cavities to the next level [1, 3].

Thermal Vapor Diffusion

To this day, the state-of-the-art growth method for producing high quality Nb_3Sn cavities in a reproducible fashion

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remains thermal vapor diffusion [2]. This technique involves exposing a niobium cavity to vaporized tin and tin chloride (which serves as a nucleating agent) in a high temperature vacuum furnace. Inspired by the Wuppertal method, at Cornell University we incorporate the use of a secondary heater for the tin source, and we impose a temperature gradient between the tin crucible and the cavity during coating. A temperature profile of a Nb_3Sn coating run at Cornell is shown in Fig. 1, and it consists of 3 main stages: nucleation, coating and annealing. During the nucleation stage, both the cavity and the $Sn/SnCl_2$ source are kept at 535 °C for 5 hours, during which tin-rich droplets start to form on the niobium oxide. Then, the temperature of the tin heater is ramped up to 1405 °C, while the cavity is kept at 1156 °C for 1.5 hours for coating. Lastly, we turn off the secondary (tin) heater and anneal the cavity at 1156 °C for one hour.
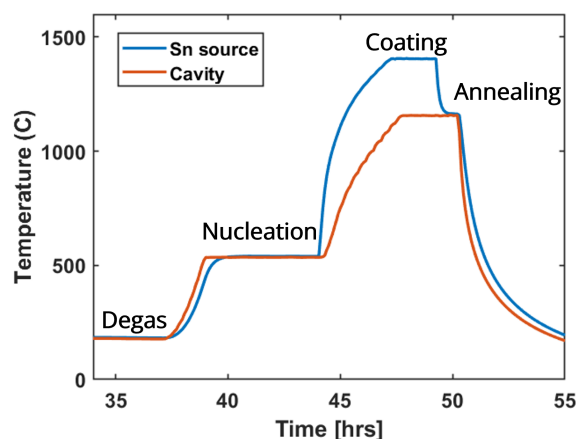

Figure 1: Vapor diffusion temperature profile at Cornell University showing the temperatures of the $Sn/SnCl_2$ source and Nb substrate (cavity) versus time. The main three stages of coting are nucleation, coating, and annealing.

Limitations

There are several practical limitations that stand in the way of Nb_3Sn cavities reaching the predicted fundamental limits of this material. It is very challenging to grow a smooth layer of stoichiometric Nb_3Sn with uniform thickness and composition. Deviating from the 3:1 atomic percent ratio of Nb to Sn in stoichiometric Nb_3Sn can result in drastic drop in both critical temperature and energy gap [5–7], which results in higher surface resistance and limits RF performance.

Not only does the layer composition need to be uniform, but so does the thickness. Due to this material's significantly lower thermal conductivity, a thicker layer will be thermally

unstable and cause a premature quench in the presence of defects. On the other hand, if the layer is not thick enough (we aim for the layer to be many penetration depths thick, so roughly at least 1 μm thick everywhere), we risk exposing regions near the Nb-Nb$_3$Sn interface which are tin depleted [2, 8, 9].

Another issue which needs to be addressed is surface roughness, which can cause loss of superconductivity due to field enhancement (at surface peaks) and magnetic flux penetration prior to reaching the superheating field [2, 10].

Decreasing the Nb$_3$Sn film thickness and surface roughness could potentially increase the quench field of our cavities [2]. Indeed, the current world record for Nb$_3$Sn quench field of 24 MV/m achieved at Fermilab was achieved via a thin (≈1 μm), smooth Nb$_3$Sn film [11].

There is continuous effort and progress in addressing these issues and improving the growth of Nb$_3$Sn. The main two paths forward are improving the vapor diffusion method and exploring alternative growth methods. This paper will give an overview of the latest developments of higher performance Nb$_3$Sn cavities at Cornell University.

IMPROVING THE VAPOR DIFFUSION METHOD

The first path forward in addressing the limitations of current Nb$_3$Sn films is improving the diffusion-based growth technique.

Thin Film Cavity

Thermal instability has been identified as a major component in SRF cavity quench, making good thermal conductivity a necessary component in achieving higher accelerating gradients. The thermal conductivity of Nb$_3$Sn is almost three orders of magnitude lower compared to that of niobium [12, 13], which can cause issues with thermal stability and thermal feedback: the heating of the Nb$_3$Sn layer in the presence of a defect will cause the surface resistance to increase, which will cause more heating and bring the cavity in a thermally unstable regime. This makes Nb$_3$Sn cavities sensitive to small, localized sources of increased dissipation, e.g., due to normal conducting defects [2]. Thermal simulations have shown that decreasing the layer thickness will increase the stability of the Nb$_3$Sn region. For example, if the film thickness is decreased from 3 μm to 1.5 μm, then the estimated quench field increases from 73 mT to 117 mT with the same defect size of 3 μm present [2].

Furthermore, thinner films have reduced surface roughness [14], which mitigates field enhancement and increases the vortex entry field at the grain boundaries.

To this end, inspired by the shiny thin film cavity [11], a modified thin-film coating was developed at Cornell University. Figure 2 shows the thermal profile of the new coating process. The main notable changes are the earlier ramp up of the secondary heater (compared to our 'standard' recipe) and the shorter coating time, which is expected to result in a smoother and thinner (≈1.5 μm thick) film.
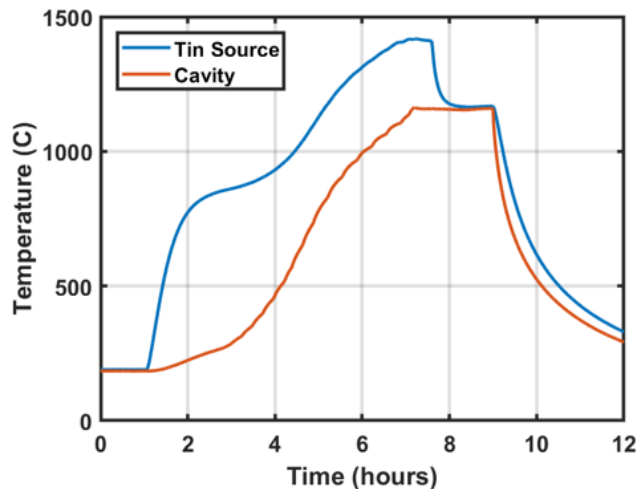

Figure 2: Vapor diffusion furnace temperature profile for thin film coating [2]. This temperature profile was inspired by the Fermilab shiny thin film coating [11]. The tin vapor pressure is increased earlier in the coating process, the 5 hour nucleation was skipped, and the coating time was decreased to achieve a thinner film.

The cavity coated had a visually shinier surface, which is interpreted as lower surface roughness. This was confirmed by a witness sample coated using the same recipe, which showed a significantly reduced surface roughness compared to the typical surface roughness of a typical coating. This can be seen in the amplitude spectral density plot of the new vs standard coated sample shown in Fig. 3.
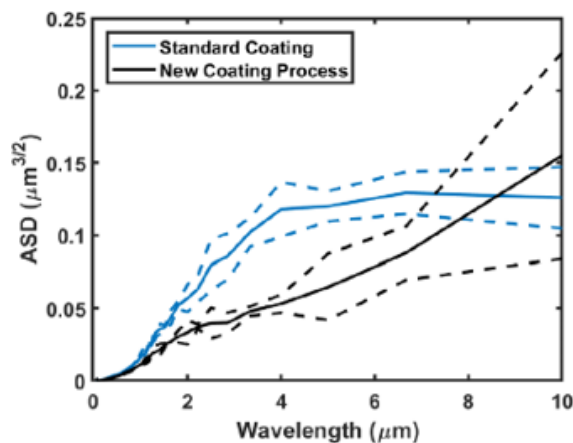

Figure 3: Amplitude Spectral Density (ASD) of witness sample coated with the thin film coating thermal profile shows reduced surface roughness in comparison to the standard coating.

While this attempt shows promise, there is still room for improvement. The Cornell thin-film cavity showed low BCS resistance, but high residual resistance. Optical inspection showed signs of what we suspect to be patchy regions with non-uniform surface composition and roughness. These types of defects are associated with larger residual resistance [2]. Unfortunately the cavity quenched at similar fields to our standard coatings.

While thin film coatings are very promising to push the accelerating gradient further, the key challenge is consistently producing a uniform layer. Producing smooth, thin coatings is not yet an established and repeatable process.

Nucleation Study

Studies have shown that the niobium oxide plays an important role in the binding of $SnCl_2$ (the nucleating agent) to the niobium surface during nucleation [15, 16]. It is well-established that anodizing the niobium surface prior to coating results in a more uniform distribution and higher density of tin-rich droplets during the nucleation stage, which also corresponds to a finer grained, smoother final film [2, 8].

This inspired Density Functional Theory (DFT) calculations [15, 17] which calculate the binding energy of $SnCl_2$ molecules to niobium oxide layers with a lack of and excess of passivating OH groups on the surface. The results suggest that it is more favorable for $SnCl_2$ to bind to both the OH poor and OH rich oxides, rather than to the neutral native oxide. The most $SnCl_2$ binding sites, which are correlated to a more uniform nucleation, are predicted to be ones created by acidic solutions which remove OH groups.
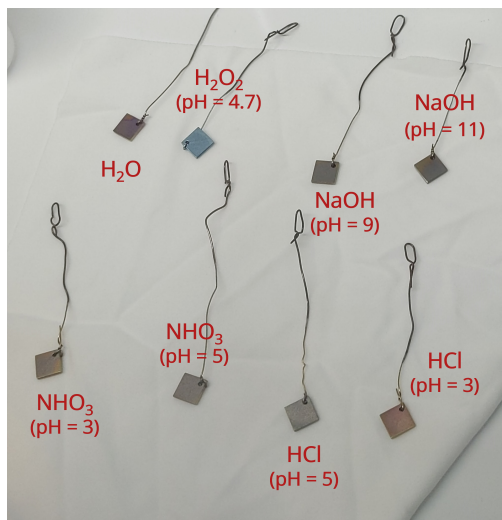

Figure 4: Photo of samples after soaking in chemical treatments. From top down, left to right, the chemical treatments (and their corresponding pH values) are: H_2O, H_2O_2 (pH = 4.7), NaOH (pH = 9), NaOH (pH = 11), NHO_3 (pH = 3), NHO_3 (pH = 5), HCl (pH = 5), and HCl (pH = 3). Control sample is not shown.

Our goal is to investigate if and how various chemical treatments with a range of pH values affect the nucleation stage of vapor diffusion based growth. We aim to find an optimal treatment which will promote dense and uniform distribution of nucleation sites, and consequently a smoother and more uniform layer than the current recipe. This study presents the results of a developed framework which quantitatively compares the impact of surface chemistry on the binding of $SnCl_2$ to the niobium oxide surface through a sample study [18].

Nine samples were electropolished and anodized prior to being soaked for 30 minutes in chemical treatments with various pH values ranging from 3 to 11 in a nitrogen glovebox. Figure 4 shows a picture of the samples after the chemical treatments, with each treatment (and respective pH value) labeled. From top down, left to right, the chemical treatments (and their corresponding pH values) are: H_2O, H_2O_2 (pH = 4.7), NaOH (pH = 9), NaOH (pH = 11), NHO_3 (pH = 3), NHO_3 (pH = 5), HCl (pH = 5), and HCl (pH = 3). The control sample (not shown in picture) was not soaked in any chemical treatment post anodization. After the chemical treatments, the samples were loaded in the vapor diffusion furnace and the furnace run was stopped after the nucleation step [18]. Samples were then extracted for surface imaging and characterization.

The surface characterization techniques used in this study include Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). The SEM images were processed and analyzed to investigate the formation of nucleation sites (tin-rich droplets). The two metrics extracted from SEM are statistics of the density of droplets (over many $\approx 16\,\mu m^2$ areas) and the average distance to each droplet's six nearest neighbors.

The results are discussed in detail in [19]. The primary finding worth highlighting is that the H_2O_2 treatment demonstrated the lowest density of non-uniformly distributed droplets. We also identify promising treatments such as NHO_3 with a low pH value.

Knowing that Nb_3Sn grains have a roughly 1 μm radius, we predict that having at least one nucleation site per 1 μm^2 area will result in a uniform and dense nucleation. Figure 5 illustrates the assessment of each sample to this criterion. Specifically, the plot shows the normalized number of imaged SEM regions with a density of less than 1 μm^2 in the blue histogram, and the normalized number of droplets with average distance to six nearest neighbors being larger than 1 μm in the red histogram.

The treatments that stood out are NHO_3 with the lowest pH value and NaOH with the highest pH value, both having no areas with density lower than 1 μm^2. The NHO_3 treatment samples showed the lowest number of average distance to six nearest neighbors being larger than 1 μm. H_2O_2 had the most regions with insufficient nucleation based on both metrics.

EDS analysis was conducted on the samples to determine the atomic composition of tin and niobium on the surface. By keeping the accelerating voltage constant, we obtained a relative comparison of the ratio of Sn to Nb atomic composition among our samples. We probed isolated droplets and areas between the droplets using point analysis scans as illustrated in Fig. 6.

The results are shown in Fig. 7. We observed significant variation in the Sn:Nb atomic percentage ratio among the droplets probed. This was not correlated to the size of the droplets. We confirm the presence of tin in areas with no droplets present for all the treatments, with the exception of H_2O_2 treated sample which consistently showed no traces
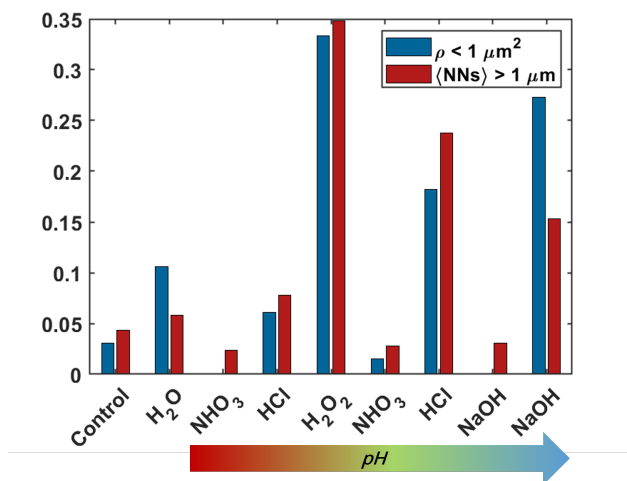
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Figure 5: Normalized counts of SEM images analyzed which have a density of under 1 nucleation site per /μm^2 are shown in blue histogram (left). Normalized counts of all doplets which have average distance to six nearest neighbors greater than 1 μm are shown in red histogram (right).
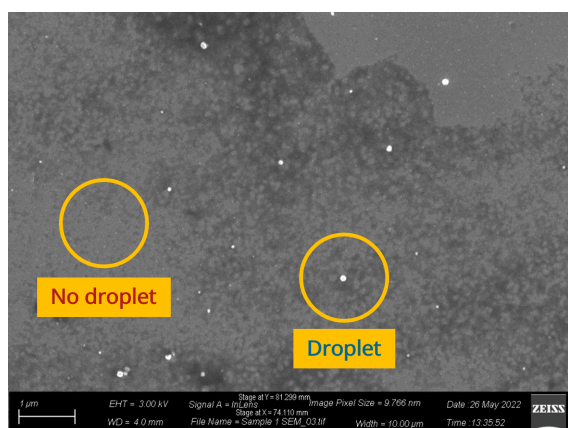

Figure 6: SEM image of H_2O_2 treated sample. Circled areas demonstrate roughly the probed area by an EDS point analysis scan for what we refer to as 'droplet' and 'no droplet' scans.

of tin when there were no droplets present. We conclude that H_2O_2 treatment suppresses $SnCl_2$ nucleation, unlike the other treatments with similar pH values.

From these findings, it is clear that a combination of both surface imaging and elemental composition is necessary to gain information about the quality of nucleation. We see that chemical treatments of the niobium substrate pre-coating influence the distribution of nucleation sites. We note that the type of chemical treatment appears to have more of an impact on the quality of nucleation compared to the pH of that treatment. A promising treatment to arise from this study is NHO_3. However, we still do not understand exactly why and how each specific treatment is influencing the nucleation differently, and further investigations will be done.
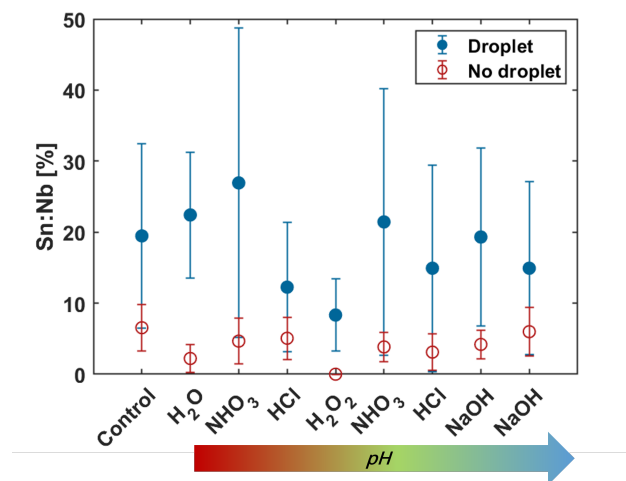

Figure 7: Atomic composition ratio of tin to niobium of droplet and non-droplet point analysis scans. Nucleation site areas are plotted in blue, while non-nucleation site areas are plotted in red hollow circles.

Future steps include performing another sample study to confirm repeatability, followed by a full coat to see how these nucleated tin-rich droplets and the tin thin film between them affect the final Nb_3Sn layer. Furthermore, we plan on applying our most promising treatments to a Nb cavity and measuring RF performance.

EXPLORING ALTERNATIVE GROWTH METHODS

Complementary to the efforts in improving the current state-of-the-art growth method, other alternative growth methods are being explored to achieve higher control of the Nb_3Sn film growth.

Electrochemical Synthesis Based Growth

A very exciting new development at Cornell University is the design and realization of a deposition process using direct electrochemical deposition which has successfully been scaled up to a 1.3 GHz cavity. This method was developed by Z. Sun [20] here at Cornell University, and the setup is shown in Fig. 8. This technique involves depositing layer of tin on the niobium substrate using seed-free electrochemical deposition, followed by high-temperature annealing to achieve thermal conversion to a smooth, stoichiometric Nb_3Sn layer. More details on the proccess can be found in [20].

Sample studies have shown low surface roughness and a good 3:1 niobium to tin atomic percentage ratio with little variation of tin content with respect to depth [20, 21]. Process was optimized for the complex geometry of a cavity which was then annealed in the vapor diffusion furnace following a typical thermal diffusion coating profile. Pictures of the cavity after the annealing process are shown in Fig. 9.

Despite the non-uniformities shown on the right picture of Fig. 9, the RF performance of this cavity was outstanding.
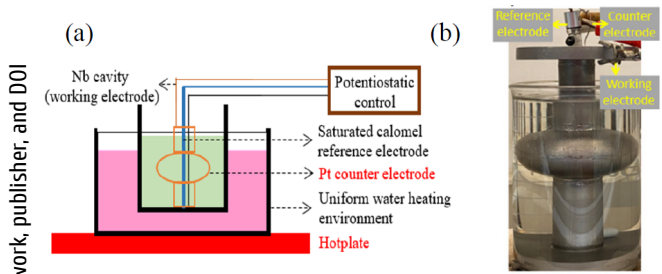
Figure 8: Schematic (a) and picture (b) of electroplating setup at Cornell University. Picture was taken from [21]. Following the electrochemical deposition of a tin layer, the niobium substrate (cavity) is annealed at high temperatures to thermally convert the deposited tin on the niobium to a smooth, stoichiometric Nb_3Sn layer. More details on the proccess can be found in [20].
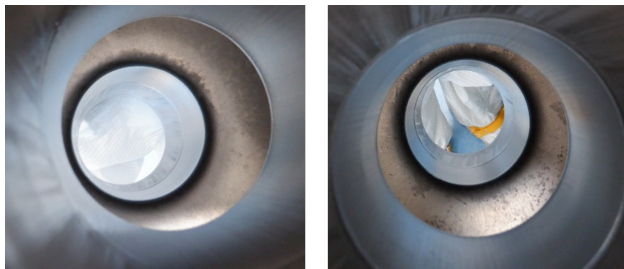

Figure 9: Pictures of electroplated cavity post-annealing. Non-uniformities seen in the right picture are due to issues with the electrode.

Figure 10 shows the quality factor vs accelerating gradient (for two different cooldowns of this cavity, labelled with C1 and C2) at 4.2 K and 2 K, plotted against a good performance vapor diffused cavity. The quality factor is above 10^{10} with a quench field of 13 MV/m. This is the first Nb_3Sn cavity produced with a method other than vapor diffusion to ever reach these quality factors and accelerating gradients.

Furthermore, analysis of the surface resistance as a function of temperature demonstrates a reduced BCS resistance for the electroplated cavity compared to a vapor diffused Nb_3Sn cavity [20].

Chemical Vapor Deposition

Another exciting development from Cornell University is the significant progress in developing a remote plasma-enhanced chemical vapor deposition (RPECVD) system [22]. This system, shown in Fig. 11, consists of a high-temperature, clean vacuum furnace which will enable a more controlled deposition process of Nb_3Sn (among other alternative SRF materials) by carefully tuning the deposition conditions for the selected chemical precursors. The use of plasma allows for deposition at lower temperatures.

The CVD furnace will explore two main methods of growing Nb_3Sn films. The first one is depositing a Sn layer on top of the Nb substrate, and then thermally convert to Nb_3Sn film through annealing, similar to the electroplating approach described in the previous section. The second approach is using both Sn and Nb precursors at high temperatures
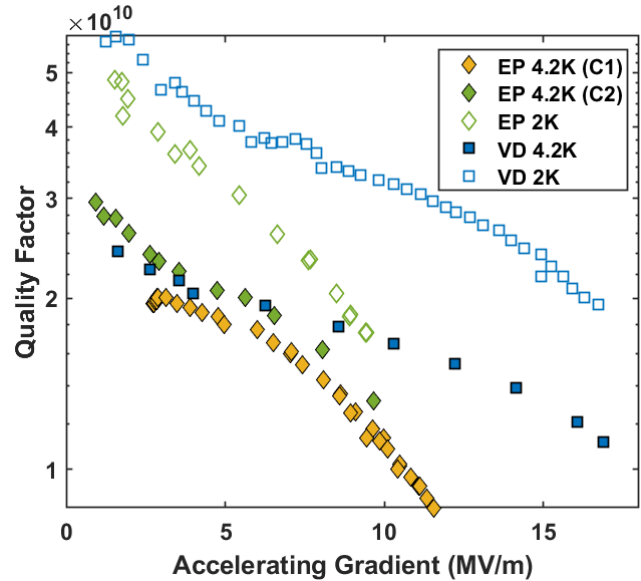

Figure 10: RF results of electroplated cavity (labeled with EP), plotted against a vapor-diffused cavity (labeled with VD) at 4.2 and 2 K. The Q vs E curves shown for the electroplated cavity at 4.2 K labeled as C1 and C2 refer to two different cooldowns. Cooldown 1 (C1) data shows the quench field of this cavity at 13 MV/m.
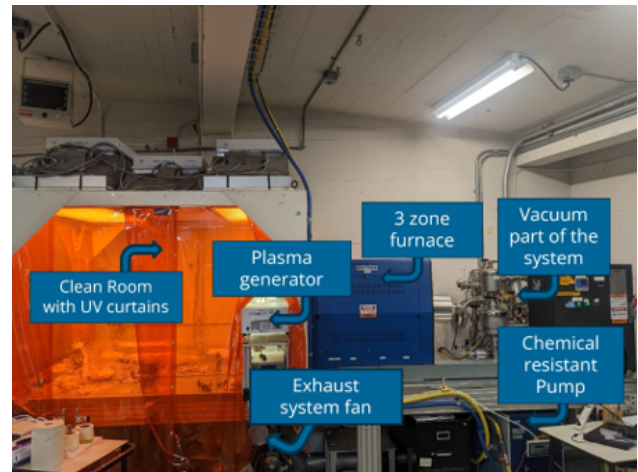

Figure 11: Setup of the remote plasma-enhanced chemical vapor deposition (RPECVD) at Cornell University.

(825-850 °C). More details on the current progress of commissioning can be found in [23]. Both methods are very exciting new fronts of possibly overcoming the limitations of the current growth methods, and even possibly taking things a step further to create a framework to coat Nb_3Sn on copper.

CONCLUSION

Nb_3Sn is a very promising material for SRF applications and is very close to reaching its first applications in the accelerator community. There are significant efforts worldwide to push the performance of Nb_3Sn SRF cavities closer to their fundamental limits, however we are limited by practi-

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cal challenges in the growth of this material. The future of Nb_3Sn is looking towards growing thinner, smoother films of uniform thickness and composition.

Cornell University's efforts to this end have focused on improving the current state-of-the-art growth method of thermal vapor diffusion. One attempt focused on altering the thermal vapor diffusion coating profile which achieved smoother films in the witness sample, but unfortunately did not have the performance we hoped for in a cavity. While smooth, thin film coatings are a very promising path to push the accelerating gradient beyond the current limits, the key obstacle is consistently producing a uniform layer, which is not yet an established and repeatable process.

Another approach to developing more uniform coatings is optimizing the first stage of growth, namely the nucleation phase. We have found that chemical treatments applied prior to coating affect the binding of the nucleating agent to the niobium surface, showing promise for more control in the diffusion-based growth of this material [19].

Additionally, we are exploring new, exciting alternative growth methods. We present very exciting RF results for the first ever successful alternative growth method of Nb_3Sn to vapor diffusion. This growth technique is achieved via direct electrochemical deposition followed by thermal annealing [20]. Furthermore, there is significant progress in commissioning a Chemical Vapor Deposition system to allow for more control over the growth process for complex geometries used in SRF applications.

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