DEVELOPMENT OF A SYSTEM FOR COATING SRF CAVITIES USING REMOTE PLASMA CVD[*]

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Abstract

Next-generation, thin-film surfaces employing Nb_3Sn, NbN, NbTiN, and other compound superconductors are destined to allow reaching superior RF performance levels in SRF cavities. Optimized, advanced deposition processes are required to enable high-quality films of such materials on large and complex-shaped cavities. For this purpose, Cornell University is developing a remote plasma-enhanced chemical vapor deposition (CVD) system that facilitates coating on complicated geometries with a high deposition rate. This system is based on a high-temperature tube furnace with a clean vacuum and furnace loading system. The use of plasma alongside reacting precursors will significantly reduce the required processing temperature and promote precursor decomposition. A vacuum quality monitor (VQM) is used to characterize the residual gases before coating. The CVD system has been designed and is currently under assembly and commissioning.

INTRODUCTION

Niobium-3 tin (Nb_3Sn) is the most promising alternative material to niobium for SRF accelerator cavities. The material has the potential to double accelerating gradients and operating temperature of SRF cavities, decreasing costs and increasing efficiency of future accelerators, see [3]- [7]. The dominant process currently used at Cornell University and elsewhere to coat Nb_3Sn films is based on vaporizing tin in a vacuum furnace and allowing the tin on the surface of an Nb substrate cavity to form Nb_3Sn. This vapor diffusion growth process creates films of good quality, but defects and surface roughness still limit these films well below the ultimate potential of this material. Exploring alternative Nb_3Sn growth methods is therefore of importance, and might offer more control over the growth process. Very thin (tens of nm) films of Nb_3Sn, NbN, NbTiN, and other compound superconductors might also promise superior RF performance levels in SRF cavities, but growing these will require advanced deposition processes to achieve high-quality, uniform films of such materials on large and complex shaped cavity surfaces. Cornell University is therefore developing a dedicated cavity CVD growth system as described in the following sections.
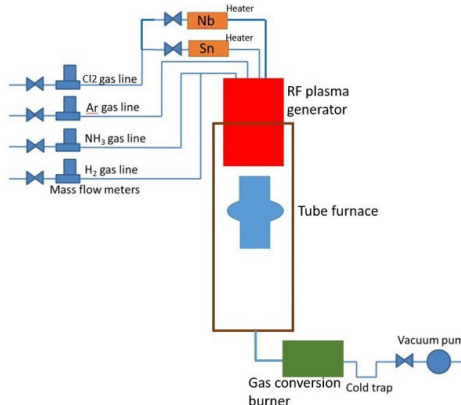
Figure 1: Schematic of the CVD system.

DESCRIPTION OF SYSTEM

The schematic of the system is shown in Fig.1 and a picture of the system in Fig. 2. The tube furnace contains a quartz tube, that can be heated safely up to 1100 C.

The furnace can be used for annealing and CVD work. A branch of the vacuum system contains a roughing pump used for CVD and another branch that contains a turbopump, which allows for smaller pressures to be achieved for annealing processes.

Near the gas input side of the tube furnace, there is an RF(Radio Frequency) plasma generator used in the plasma-enhanced CVD process.

The system contains a Vacuum Quality Monitor (VQM) for monitoring possible contamination, as it has the advantage of not over-representing hydrogen when compared to a classical RGA.

On the side for loading cavities, we have a clean room to maintain the cavities and samples free of contamination as seen in Fig. 2.

Chemical Vapor Deposition (CVD)

CVD is a vacuum deposition method and it offers a potential path to grow high-quality films of Nb_3Sn, NbN, NbTiN on various substrates including niobium and copper. An example of the use of CVD is given in [2], and a paper detailing RF results for CVD on a cooper substrate is given in [1] .

A substrate is heated up and exposed to precursors. Figure 1 shows a cavity that is heated, representing the substrate. Nb and Sn precursors decompose at the surface of the cavity and nucleate to Nb3Sn grains. The chemical equation describing the deposition of a film of Nb_3Sn is:

$$3NbCl_x + SnCl_y + (3x+y)/2 H_2 \rightarrow Nb_3Sn + (3x+y)HCl \quad (1)$$
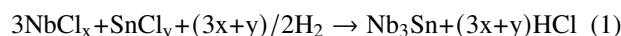
Reaction/deposition takes place on the substrate surface and leaves a thin-film coating on it. Note that Hydrogen is added for the reaction and then the resulting gasses are pumped away.

CVD has certain advantages:

- It has a high deposition rate 300 µm/hour and this allows us to make both thick and thin films.
- It is strong enough to withstand High-Pressure Rinses (HPR) and tumbling.
- It gives better control on the system and the chemistry compared with other methods.

Remote Plasma-enhanced Chemical Vapor Deposition (CVD) System

An RF generator is included in the CVD system to create plasma. The use of plasma can significantly reduce the processing temperature and promote precursor decomposition. We anticipate that this remote plasma-enhanced chemical vapor deposition (CVD) system will facilitate coating on complicated geometries with a high deposition rate.

SYSTEM COMPONENTS

The tube furnace used is a Lindberg/Blue M™ 1500 °C Heavy Duty Tube Furnace. The furnace itself has 3 zones with independent controls and can reach temperatures of 1500 C.

The quartz tube has shielding at the ends of the outer heating zones of the tube furnace, as seen in Fig. 3, to prevent the end flanges which contain silicon O-rings from getting too hot. The heat shields also help to keep the temperature uniform inside the tube furnace.

Cooper gaskets are used for most connections in the system, as this allows for bakes of the system, and reduces the possibility of leaks.

On the part of the system used for CVD work, we have a butterfly valve to control the flow of gasses and a cold trap to condense any potentially corrosive gasses.

A turbo pump is used to bring the pressure in 10^{-7} Torr range for annealing processes. The VQM is used when the turbo pump is in operation. For the CVD process, we use a roughing pump.
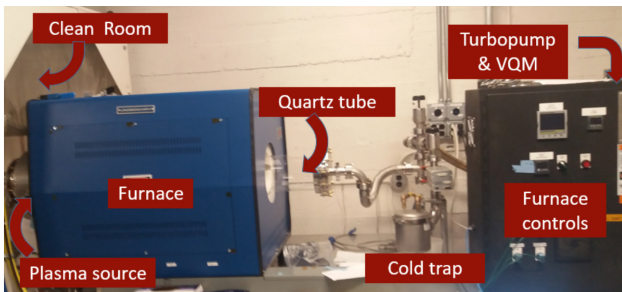

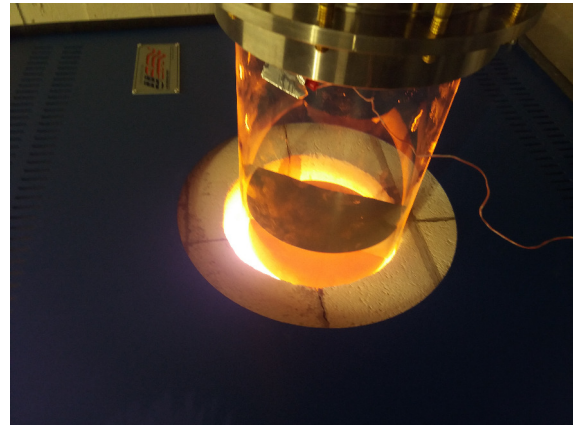Figure 2: Tube furnace system and furnace controls.

Figure 3: Tube furnace in operation.

The loading system for cavities is shown in Fig. 4 and it will be inside a cleanroom to maintain contamination at a minimum. The cavities and samples will be loaded using Molybdenum boats as in Fig. 5, as the melting temperature of Molybdenum is very high(2896 K), the thermal expansion coefficient is quite small ($4.8 \cdot 10^{-6}$ / K at 25 C).

We plan to coat and anneal samples alongside 3.9 GHz and 2.6 GHz cavities.

The RF generator will decompose the precursor gasses $NbCl_x$ and $SnCl_y$ into ions of Nb, Sn, and Cl.

Plasma Characterization

A spectrometer will be used to measure the emission of plasma ions. This will offer data on the relative concentrations of the precursors and the resulting gasses. The known spectrum of ions can be used in this characterization. This ensures the reaction is well controlled and that the precursors have the right proportions.

The temperature distribution and the presence of plasma as a function of distance will be measured using thermocouples.
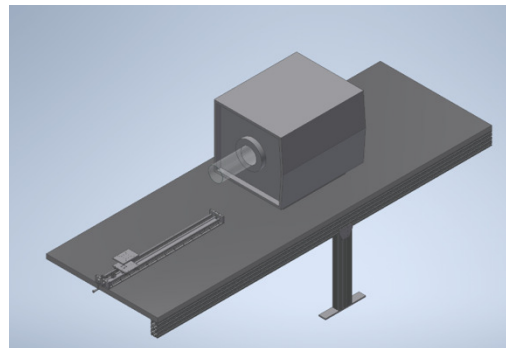

Figure 4: Loading system for tube furnace.

COMMISSIONING

Improving the Vacuum

Using the turbo-pump, the lowest pressure obtained after baking the tube furnace is $7.8 \cdot 10^{-7}$ Torr. We use Fomblin

Figure 5: Molybdenum boats.

on the O-ring seal of the flanges on the quartz tube to reduce the likelihood of leaks. The Fomblin has a low vapor pressure 10^{-12} Torr to minimize the risk of contamination. The vacuum improved significantly with bakes at temperatures high enough to allow for significant heating of the outer parts of the quartz tube.

Silicon protections were added between the quartz tube and the side flanges to prevent the quartz tube from cracking. They protect the quartz from bolt over-tightening and from the movement of the metal flanges when the air is pumped out.

Further improvement of vacuum seems possible by having longer bakes at a higher temperature, or by wrapping heating tape around the exposed quartz outside the tube furnace, to help with outgassing.
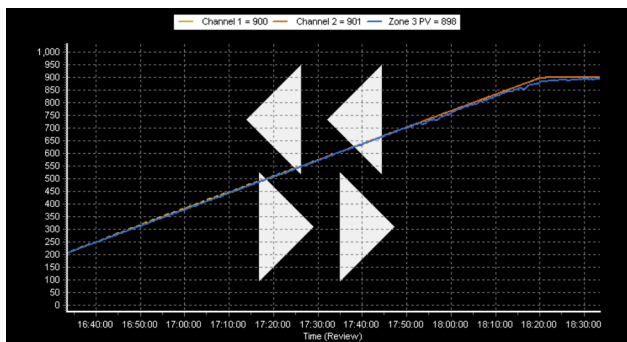

Figure 6: Temperature ramping for the 3 zones till 900 C.

Controls for 3 Zones

The furnace and its control system were commissioned successfully. Figures 6 and 7 show examples of temperature ramps of the system to 900 C and 1100 C respectively.

VQM System

Testing the VQM system revealed some peaks that seemed to originate from methanol evaporation, as at very small pres-
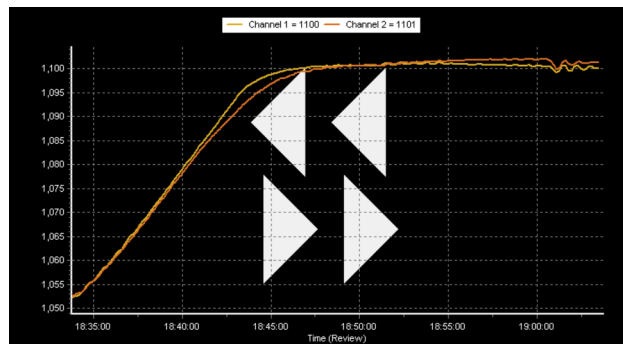

Figure 7: Raising the temperature in the furnace to 1100 C.

Figure 8: Gases and contamination in the system monitored by a VQM.

sures the dominant mass peak was 31 on the spectrum, as seen in Fig 8. Multiple bakes were performed on the mass spectrometer while isolating it from the rest of the system but the peak at mass 31 corresponding to methanol was still present. After discussions with the technicians responsible for the VQM system, it was deduced that the erroneous values might come from some hydrocarbon contamination. The whole system was cleaned again and reassembled, replacing most of the CF flanges with KF flanges.

FUTURE STEPS

We are currently installing a venting system with filtered nitrogen to keep the furnace clean. Niobium samples will be annealed to check the cleanliness of the furnace before proceeding further. Finding an appropriate spectrometer to characterize the plasma ions is also an important next step to allow for accurate measurement of the proportions of ions produced during precursor decomposition. This will allow for control of the chemistry and help avoid the production of unwanted chemical compounds. Then, the next step is the conversion of electroplated Sn on Nb to Nb_3Sn on samples and high-frequency cavities, followed by first studies on CVD of Nb and Sn.

REFERENCES

[1] M. Ge *et al.*, "CVD Coated Copper Substrate SRF Cavity Research at Cornell University", in *Proc. 19th Int. Conf. RF Superconductivity (SRF'19)*, Dresden, Germany, Jun.-Jul. 2019, pp. 381–386. doi:10.18429/JACoW-SRF2019-TUFUB8

[2] P. Pizzol *et al.*, "CVD Deposition of Nb Based Materials for SRF Cavities", in *Proc. 7th Int. Particle Accelerator Conf. (IPAC'16)*, Busan, Korea, May 2016, pp. 2241–2244. `doi:10.18429/JACoW-IPAC2016-WEPMB056`

[3] S.Posen and M.Liepe, "Advances in development of Nb3Sn superconducting radio-frequency cavities," Phys. Rev. ST Accel. Beams, vol. 17, p. 112001, Nov. 2014. `doi:10.1103/PhysRevSTAB.17.112001`

[4] S. Posen, M. Liepe, and D. L. Hall, "Proof-of-principle demonstration of Nb3Sn superconducting radiofrequency cav- ities for high Q0 applications," App. Phys. Lett., vol. 106, p. 082601, 2015. `doi:10.1063/1.4913247`

[5] Posen and D. L. Hall, "Nb3Sn Superconducting Radio frequency Cavities: Fabrication, Results, Properties, and Prospects," Superconductor Science and Technology, vol. 30, no. 3, p. 33004, Jan. 2017. `doi:10.1088/1361-6668/30/3/033004`

[6] S. Posen et al., "Advances in Nb3Sn superconducting radio-frequency cavities towards first practical accelerator applications," Superconductor Science and Technology, vol. 34, no. 2, p. 025007, Jan. 2021. `doi:10.1088/1361-6668/abc7f7`

[7] U. Pudasaini et al., "Recent Results From Nb3Sn Single Cell Cavities Coated at Jefferson Lab," in Proc. SRF'19, Dresden, Germany, July 2019, paper MOP018, pp. 65-70. ISBN: 978-3-95450-211-0 `doi:10.18429/ JACoW-SRF2019-MOP018`