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DEVELOPMENT OF A CVD SYSTEM FOR NEXT-GENERATION SRF CAVITIES *

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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. The system can also be used for annealing cavities after the CVD process to improve the surface layer. The chlorine precursors have the potential to be corrosive to the equipment and pose specific safety concerns. A MATLAB GUI has been developed to control and monitor the CVD system at Cornell.

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 Refs. [1–5]. A vapor diffusion-based growth process for Nb_3Sn has given the best RF performance to date, but maximum fields are still well below the ultimate predicted limit of this material (24 MV/m reached vs. 90+ MV/m ultimate limit). Defects (e.g., small tin-depleted regions) and surface roughness are limiting factors of these films.

Exploring alternative Nb_3Sn growth methods is therefore one direction for improving performance beyond current limits, 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. Growing these will require advanced deposition processes to achieve high-quality, uniform films of such materials on large and complex shaped cavity surfaces.

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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 Ref. [6], and a paper detailing RF results for Nb CVD on a cooper substrate is given in Ref. [7]. Cornell successfully tested first-ever fabricated CVD Nb_3Sn-on-copper SRF cavities [8]. These are comprised of copper substrates with thin-film interior surface coatings of niobium interlayer/CTE(coefficient thermal expansion)-bridge and Nb_3Sn formed via chemical vapor deposition (CVD). The coating was performed by industry partner Ultramet using unique CVD precursor materials developed by researchers at Florida State University. Ultramet observed a Sn concentration of 24-25 % on coupon samples. A critical temperature $T_c \approx 16.5$ K suggests that the uniformity of the Sn to Nb ratio needs to be improved on the cavity surface. The RF performance was limited by high residual resistance, likely due to contamination or defects on the surface. Further improvement in the coating process will be needed to improve RF performance.

Cornell University is therefore developing a dedicated cavity chemical vapor deposition growth system as described in the following sections.

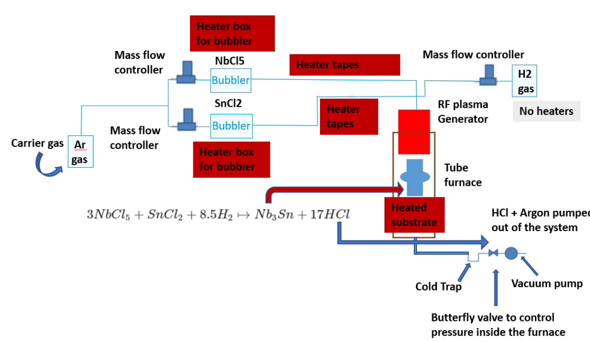

Figure 1: Schematic of the CVD setup at Cornell.

DESCRIPTION OF SYSTEM

The schematic of the system is shown in Fig. 1 and a picture of the system in Fig. 2. The basic layout and design has been described before in Ref. [9], so this paper will focus on recent progress and development.

The furnace has been used for annealing coupon samples and is now being developed to be ready for Nb_3Sn CVD. A branch of the vacuum system contains a roughing pump

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used for CVD and another branch contains a turbo-pump and an ion pump, which allows for smaller pressures to be achieved for the annealing processes.

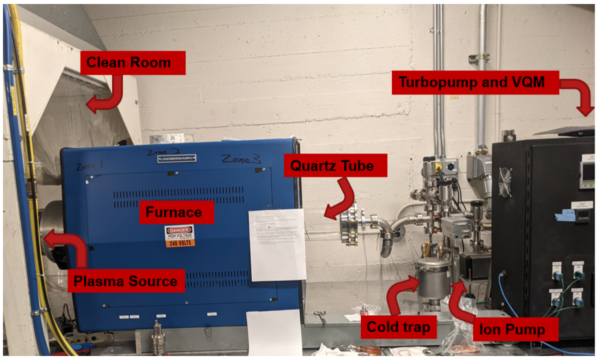

Figure 2: Tube furnace system and furnace controls.

Design and Safety Considerations

Figure 1 shows the basic process of the CVD system. The cavity is heated by the furnace, representing the substrate of the chemical reaction. Nb and Sn precursors react with hydrogen gas and decompose at the surface of the cavity to form Nb_3Sn grains. The chemical equation describing the deposition of a film of Nb_3Sn is:

$$3NbCl_5 + SnCl_2 + (15+2)/2H_2 \longrightarrow Nb_3Sn + 17HCl. \quad (1)$$

Reaction/deposition takes place on the substrate surface and leaves a coating on it. The corrosive and unreacted gasses (HCl, $NbCl_5$, $SnCl_2$) are condensed using a cold trap to protect the butterfly valve, the pressure sensor and the roughing pump from damage.

The solid precursors $NbCl_5$ and $SnCl_2$ will be used for the initial CVD Nb_3Sn run. Both can corrode stainless steel when exposed to humidity, so hastelloy bubblers will be used to eliminate the danger of corrosion. If $SnCl_2$ is not volatile enough, other precursors can be used (like $SnCl_4$), but $SnCl_2$ has the advantage of being relatively safe to use compared to other precursors. $SnCl_4$ has a higher vapor pressure than $SnCl_2$ at a given temperature and is used in ALD processes [10], but has added safety risks, due to its liquid form and high vapor pressure at room temperature.

A gas cylinder that contains a mixture of argon and hydrogen will be used for the hydrogen part of the chemical reaction and argon will be used as the carrier gas. One gas cylinder containing both the argon and hydrogen has the advantage of reducing the fire hazards associated with pure hydrogen.

Safety considerations related to the concentration of $SnCl_2$, $NbCl_5$ and HCl are taken into account in developing clear safety guidelines and procedures. There will be sensors for detecting dangerous leaks of corrosive gasses like HCl gas and asphyxiating gases like argon. Figure 4 summarises the typical response to various problems that could occur in the system to ensure safe operation.

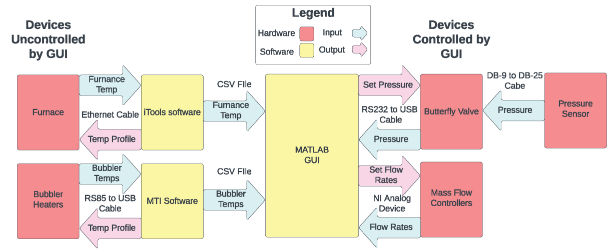

Figure 3: CVD GUI Flowchart showing communication and control for equipment.

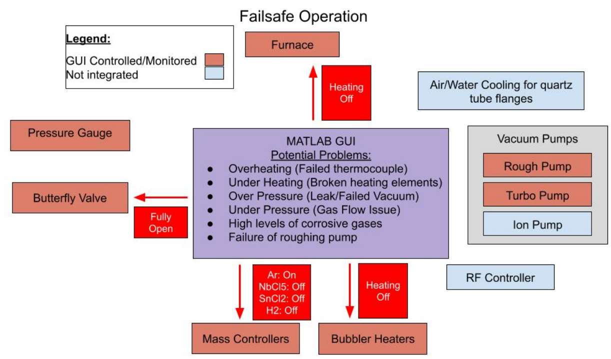

Figure 4: Fail-safe operation.

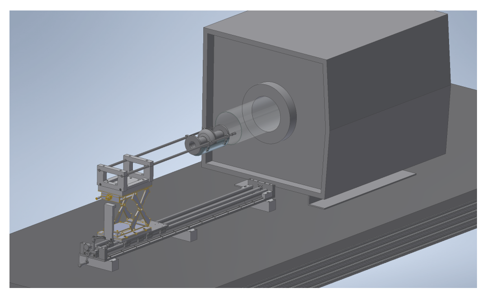

Figure 5: Schematic of the loading system for the tube furnace.

RECENT PROGRESS

A Graphical User Interface(GUI) has been designed to control the mass flow controllers, the butterfly valve, the bubbler heaters, to monitor the furnace temperature and the pressure inside. A summary of the communication and control is shown in Fig. 3.

The furnace heating is controlled by its own furnace software, but the main control GUI synchronises all the heating and flow/pressure controls and monitors the furnace operation. The temperatures in each heating zone are read and displayed in the controlling GUI.

NI input-output devices will be used for sending and reading analog signals. The flow of the mass flow controllers will be set with the help of NI devices and the roughing pump will be monitored and controlled in this way as well.

The butterfly valve is used for controlling the pressure inside the reaction chamber and is controlled remotely by the GUI using a RS 232 connection. It together with the cor-

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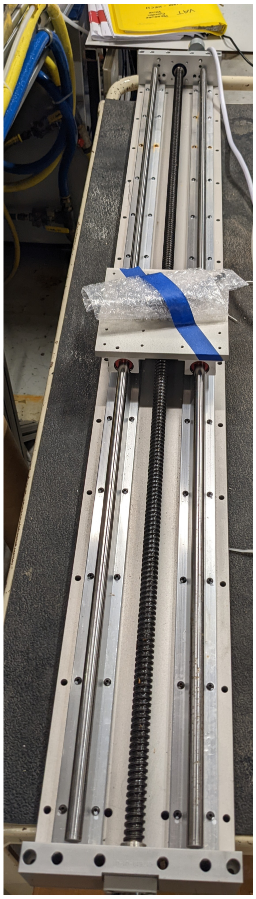

Figure 6: Loading system in assembly.

responding pressure sensor were installed and successfully tested.

The bubbler heaters are controlled remotely using a RS 485 connection. During annealing the pressure and chemical composition can be monitored using the Vacuum quality monitor and its associated pressure gauge.

Another graphical user interface was designed for the annealing process, used for monitoring the turbopump and the temperature of the furnace. The turbo-pump will be monitored using a RS 485 connection. The annealing vacuum was improved by adding an ion pump, which together with the turbo-pump improved the pumping capacity of the system. The O-ring material used in sealing the quartz tube flanges was also changed, which reduced the nitrogen diffusion though the O-ring material. The current operating pressure during annealing is in the range of low 10^{-7} Torr range which is 100 times better than when the system was first assembled.

The loading system for cavities is shown in Fig. 5. The current state of assembly is shown in Fig. 6. The cavities and samples will be loaded using Molybdenum boats.

FUTURE STEPS

In the near future, the plans are to finish the safety analysis and implement the necessary sensors and safety procedures for corrosive/asphyxiating gases. The NI multi-function input/output devices need to be installed to control and monitor the roughing pump and the mass flow controllers. The RF system needs its own testing for successfully creating plasma and insuring safe operation. The next step would be to run the first coupon samples and start parameter optimization for the CVD process. One idea is to start with just Nb CVD or Sn deposition [10], before moving towards the more complex Nb_3Sn deposition.

ACKNOWLEDGEMENTS

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