Buffer Chemical Polishing and RF Testing of the 56 MHz SRF Cavity

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Introduction:

The 56 MHz cavity presents a unique challenge in preparing it for RF testing prior to construction of the cryomodule. This challenge arises due to the physical dimensions and subsequent weight of the cavity, and is further complicated by the coaxial geometry, and the need to properly chemically etch and high pressure rinse the entire inner surface prior to RF testing. To the best of my knowledge, this is the largest all niobium SRF cavity to be chemically etched and subsequently tested in a vertical dewar at 4K, and these processes will be the topic of this technical note.

Chemical Processing:

The chemical processing of the cavity will take place at the BNL/Advanced Energy Systems (AES) chemical processing facility, located at the AES site in Medford NY, just down the road from BNL. This facility was built for the purpose of processing SRF cavities, and the equipment that was ordered was designed to accommodate the 56 MHz cavity. A schematic overview of the facility is given in figure 1, and is made up of several key components that will be described in this tech note including the ultrasonic cleaning station, the Buffer Chemical Polishing Cabinet (BCP) and the High Pressure Rinse (HPR) system.

Figure 1. The Overall SRF Cavity Processing Facility Overview
Once the cavity fabrication is completed and preliminary bench-top RF measurements and dimensional checks on a coordinated measuring machine (CMM) are finished, the cavity will begin its first pass through the chemical processing cycle. Figure 2 shows a flow diagram of the chemical processing sequence that will be carried out on the cavity and the possible different paths that will be taken. Regardless of the path, the process will start out with a degreasing of the cavity in an ultrasonic tank using a solution of de-ionized water (DI) and micro-90, a chemical surfactant commonly used on niobium cavities. Following the ultrasonic cleaning, the cavity will be passed through two DI rinse tanks to ensure removal of surfactant residue. The cavity will then be transferred to the chemistry room to begin the chemical etching of the cavity.

Figure 2. Flowchart of SRF cavity processing events
The chemical etching processes will be carried out in a buffered chemical polishing (BCP) cabinet specifically designed for SRF cavities, shown in figure 3. The BCP mixture is the traditional 1:1:2 ratio of Hydrofluoric acid (49%), Nitric acid (69.5%), and Phosphoric acid (85%) and is used to etch away the surface layers on the inside of the cavity. The chemical process involves the reaction of the nitric acid with the niobium to form a Niobium pent-oxide ($\text{Nb}_2\text{O}_5$) which then reacts with the Hydrofluoric acid to form a hydro-soluble Niobium Fluoride ($\text{NbF}_5$) which is subsequently swept out of the cavity as the acid is circulated. [1] The Phosphoric acid serves as a buffer to stabilize the rate of reaction, which is also a function of the temperature of the acid solution and the quantity of dissolved Nb present in the acid.

Figure 3. A schematic representation of the BCP cabinet that will be installed at AES.

The first time the cavity is etched the goal is to remove impurities in the surface layers which are a result of niobium sheet forming process, as well as the machining and welding steps needed to fabricate the cavity. The initial chemical etching will remove approximate 150 $\mu$m of material in two stages, with an initial 75 $\mu$m removal followed by
a 180 degree rotation of the cavity and a second 75 μm removal. The cavity is rotate to help ensure uniform material removal across the entire cavity by minimizing differences in removal rates due to changes in the acid flow pattern and temperature variations of the acid as it passes through the cavity. For all subsequent BCP processing steps the cavity will be given a light BCP, 20 μm material removal, to expose a fresh RF surface and ensure removal of any possible contaminants.

Following the chemical etching the cavity will be rinsed with 18 MOhm DI water by first filling and emptying the cavity 10 times to remove the bulk of the acid and terminate the chemical reaction. This will be followed by rinsing the cavity while monitoring the resistivity of the discharge water. The cavity will be rinsed until the discharge water measures 17.5 MOhm, ensuring complete removal of all acid. At this stage the cavity will be removed from the BCP cabinet and transferred to the high pressure rinse (HPR) cabinet.

The HPR system was also built to accommodate this cavity and is shown in figure 4. The HPR is located in a class 100 clean room to help minimize the introduction of particulate matter in the cavity. The system is designed to provide rinsing of the internal volume of the cavity with DI water at a pressure of up to 1500 psi and a flow rate of 5 gallons per minute. The system is designed with a high pressure rinse head that can be configured as needed, attached to a wand on a robotic arm. The wand enters the cavity from the top and sprays the inside of the cavity while the cavity rotates on a turntable. The rinsing duration, wand travel speed and turntable speed are all user controlled variables.

In addition the system is designed to allow for the wand to be moved off-axis up to 9 inches to allow for rinsing of the outer section of the cavity coaxial structure, a function that disables the turntable to avoid damage to the cavity. The plan is for the cavity will spend a total of 6 hours on the HPR system, with 3 hours for rinsing the center conductor region and 30 minutes for rinsing through each of the 6 cleaning ports located on the outer section of the coaxial cavity. Once the cavity rinsing is complete a hot nitrogen purge will be introduced through the HPR wand to dry the inside of the cavity. This is done to minimize the amount of time necessary for the cavity to air dry, as well as reduce the potential for particulate matter to attach itself to the wetted cavity surface.
Following the HPR cycle the cavity will be transferred to the class 10 clean room area to finish drying. At this point there is an additional step that is introduced only after the bulk BCP which is the UHV 600° Heat Treatment discussed below, for all other light BCP treatments the cavity will be partially assembled and again placed on the HPR system to rinse only the inner coaxial section for 3 hours. After this second rinse the cavity will again be moved back to the class 10 area, allowed to finish drying and the assembly will be completed. Once assembled the cavity will be attached to a dry turbo pump vacuum station, evacuated and leak checked using a residual gas analyzer and calibrated leak. Once the cavity is found to be leak tight it will be stored under vacuum and readied for transfer to BNL for cryogenic testing in our Vertical Test Facility (VTF).

**UHV 600° Heat Treatment:**
Following the bulk BCP treatment and subsequent HPR the cavity will be double bagged in anti-static clean room bags and transferred to BNL for heat treatment in our
UHV furnace. The cavity will be baked at 600°C for 10 hours to help remove hydrogen gas that is interstitially dissolved in the niobium crystalline structure. [2,3] The importance of this step is to reduce the potential for Q-disease, which is the reduction of the Quality factor, Q, of the cavity due to the precipitation of hydrogen in the niobium during cool-down for cryogenic testing and operation. The exact source of the hydrogen in the bulk Nb is a subject of some debate, as it is thought that the BCP and HPR process may introduce hydrogen gas into the material, while other data suggests that the high affinity Nb has for hydrogen is the cause, not the chemistry. Regardless of the cause, a very interesting measurement has found that regardless of where the hydrogen originates, it is important to first carry out the bulk BCP and HPR to remove the surface impurities, as failure to do so has resulted in material which showed a strong degradation in RRR value as a result of UHV vacuum baking without first carrying out the bulk BCP.[4]

The recipes for baking vary slightly, ranging in temperature from 600 to 800°C, and time at temperature from 4 to 12 hours. Regardless, the general principle is the same. The choice of 600°C is based on past success at this temperature and time combination as well as to avoid potential partial annealing of the niobium material at 800°C.

Following the UHV baking the cavity will again be placed through the chemistry processes listed above using the light BCP removal and culminating in an evacuated cavity that will be ready for RF testing.

RF Testing:

Once the cavity arrives at BNL it will be placed inside of our RF cavity work room for attachment to the test stand insert. The room is designed to allow for cavity maintenance work to be carried out, along with providing a class 100 clean environment for attachment of cavities to the test stand insert. Once the cavity is attached and RF and vacuum connections are made, a final leak check will be carried out prior to transferring the cavity into the dewar for testing. The dewar that will be used for testing the 56 MHz cavity is designed to accommodate cavities up to 38” in diameter and up to 8’ in length.

The Vertical Test Facility(VTF) will have a control room that is set up with several low level RF systems designed to drive cavities at frequencies ranging from 56 MHz up to 1300 MHz, and will have the ability to point at one of two dewars that are available for operation down to 1.8K. In addition there is a liquid helium refrigerator that has 360 W of cooling capacity at 4.45K, and a 1000 gallon storage dewar, as well as a liquid ring pump for operation down to 1.8K for other projects.

Once the cavity reaches the desired operating temperature and the liquid level is at the pre-determined height we will begin testing of the cavity. The low level RF system will be designed as a phase lock loop and will drive the high power amplifier, capable of providing several hundred watts of power to the cavity. This will be interfaced to a computer control system for data collection and logging. Following the cable calibrations the cavity will be excited and the quality factor (Q) as a function of gradient (Eacc) will be measured. During the test potential multi-pacting barriers will be processed, radiation levels monitored to ascertain potential field emission problems, and liquid helium consumption monitored. Once the testing is complete further data analysis will ensue to study the peak electric and magnetic fields reached as well as to understand the power losses measured and determine their cause. If the first test does not yield the
desired results, either due to excessive field emission, or poor performance and lower than expected Q values, the cavity will be re-processed and tested again.

References: