

STUDY OF I-V CHARACTERISTICS AT DIFFERENT LOCATIONS INSIDE A DEMOUNTABLE NB CAVITY DURING VERTICAL BEP AND EP TREATMENTS

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Abstract

For a normal superconducting radio frequency (SRF) cavity, it is hard to obtain detailed information of an electropolishing process. So, a demountable cavity was firstly made by JLab to resolve this problem. This paper reports the measurements of I-V characteristics at three different locations inside the demountable cavity during buffered electropolishing (BEP) and electropolishing (EP) treatments. The polishing plateau appeared earlier on the surface areas close to iris and later on those near equator. To find the reason for this phenomenon, the electric field distribution in the cavity was considered and simulated by means of Poisson-Superfish. Correlations were found between the measured I-V characteristics and the simulated results. This implies that electric field distribution inside a SRF cavity has an important effect on the polishing processes during vertical BEP and EP.

INSTRUCTION

Surface condition plays a critical role in the performance of a SRF cavity [1]. Since SRF niobium cavities are born, researches on surface treatment for SRF cavities have not been stopped. As the technique of surface treatments on Nb SRF cavities evolved from buffered chemical polishing (BCP) to electropolishing (EP), the performance of SRF cavities improved greatly [2-5]. The performance of SRF cavities improved greatly. However, the yield of the nine-cell cavities surpassing the required 35 MV/m for the ILC is still not high. The variability of cavity performances has emerged as a major challenge. On the other hand, the low polishing rate about 0.38/min of EP is another problem. Usually a thickness of 150 μ m is needed in order to remove the surface damage layer of Nb. Therefore, a method for SRF cavity treatment called buffered electropolishing (BEP) is developed by Jefferson Lab and have shown some advantages in small sample experiments, such as a much smoother niobium surface (about 50nm) and faster polishing (2.5 μ m/min) [6, 7]. Thus, further research of BEP on single cell SRF niobium cavities is launched.

As a R&D work, study is firstly focused on the anode polarization characteristics by employing a JLab home-made demountable cavity. With the help of small button samples on the demountable cavity, the characteristics at different locations of the cavity are obtained during BEP

process. Experimental results shows that the reaction processes at different locations may be different if an unsuitable work point is chosen for BEP. In the EP process, the similar tendency also appeared but not as obvious as that in BEP. The reasons for the difference of the reaction at different locations are analyzed. It could be understood as due to the different resistance of electrolyte for the different locations because of the variation in distance from cathode. However, further simulation with Poisson-Superfish reveals that the distribution of electric field can help us better understand this phenomenon and provide a method to resolve it.

EXPERIMENTAL SETUP

For the study, a vertical electropolishing system is built. Since it is home-made, we will do a little more introduction to this setup. As shown in figure 1, it mainly includes four parts: 1. Demountable cavity; 2. Electrolyte circulating system; 3. Data acquisition system; 4. Cooling system. With the chemical fume hood, they constitute the whole setup. The details are introduced in the following:

The upper right image in figure 1 is a CCD picture of the demountable cavity. The whole cavity is made of two half cells and three small niobium button samples. There are three sample holders in one of half cells at locations close to equator, midway between the equator and iris, and close to iris. The niobium button samples will be mounted in the holders, and electrically insulated from the cell. In this way, it allows the measurements of I-V curves at the three locations independently. At present, a new demountable cavity with two more buttons on the other half cell has been just made. The new demountable cavity will be very helpful for the further study on the effect of gravity in the vertical polishing process.

The image in the lower middle of the figure 1 is the schematic of the electrolyte circulating system of the vertical polishing system for SRF single cell cavities. For polishing, Pump A will be turned on and Valves 2 and 4 are open. The flow rate of the electrolyte is regulated via Valve 2 with a range from 0 to 10 L/min. Valve 4 is installed to branch out the electrolyte in order to avoid labouring of Pump A. Higher flow rate is possible by opening Valve 5. Draining will be achieved by opening Valves 1 and 3 and closing Valves 2, 4, and 5. For rinsing, DI water will be introduced to the acid container. Then Valves 2 and 5 are open and Valves 1, 3, and 4 are closed. In the polishing process, dry nitrogen is introduced to mix

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with the experimentally produced hydrogen in order to lower its concentration for safety.

The schematic in the upper left corner shows the data acquisition system including two parts: hardware and software. The hardware includes following parts: a power supply manufactured by Electronic Measurements Inc. (Model # 40T250) for the cavity and three button samples; three high resolution current sensors to get the current information of three button samples; two kinds of temperature sensors to monitor the temperatures on the cavity outer wall and the electrolyte in the container; National Instruments SC-2345 signal conditioning Chassis as well as SCC-AI01 and SCC-AI06 DAQ cards for the acquisition of voltage and current signals. Then, all the measured signals including voltage, currents and temperatures will be transferred to LABVIEW

programmed software in the computer for processing and controlling.

The last part is a cooling system. It mainly includes outer and inner cooling two parts. The outer cooling is provided by a Teflon box surrounding the demountable cavity. It has two water inlets, one for cold city water and the other for hot. By adjusting the ratio between cold and hot water, the box can be filled with water of selected temperature according to the need. The inner cooling part includes a chillier and aluminium tubing. The chillier is made by Neslab Instrument Inc. with the Model #CFT-150. It can provide cooling water with the lowest temperature 5°C. The aluminium tubing is wrapped by PTFE tape and inserted to the electrolyte container for heat exchange.

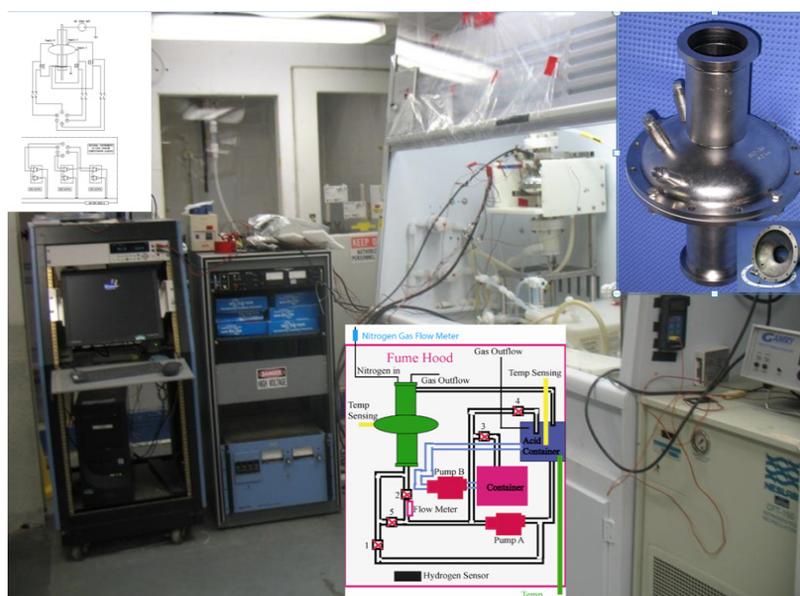


Figure 1: The picture of vertical BEP setup and the demountable cavity.

RESULTS AND DISCUSSION

Research on I-V characteristic is one of the most important methods to understand an electrochemical process. For either BEP or EP, the typical I-V characteristic curves are similar. Four typical regions in the I-V curve should appear corresponding to etching, periodic oscillation of anode current density, polishing, and gas evolution on the anode surface. However, due to the complication of the real polishing configurations, the detailed shape of each curve may be various. But on the other hand, we also could get the differences of reaction processes from those different I-V curves. Following, we will take a series of I-V curves from EP and BEP experiments as examples, which include button sample I-V curves and a cavity I-V curve, to understand the

different reaction processes at the different locations of the single cell SRF cavity.

I-V Characteristics of a Single Cell Cavity During EP Process

Figure 2 is a series of I-V curves obtained during a vertical EP process by using the demountable cavity. As shown in the first reaction region, etching region, I-V curves of the three samples begin to show the differences between each other. The slope of sample 1 is the steepest of all, and then is sample 2 followed by sample 3. It can be simply explained by the reason mentioned above that the behaviour in the region is dominated by electrolytic resistance. Since sample 1 is the closest to the cathode, the resistance between it and the cathode is the smallest. So, its slope is the steepest, while the slope of sample 3 is

the least steep. In the later of this paper we will discuss this further.

After the etching region, the I-V curve comes to its oscillation region. Although in the etching region, the process mainly follows Ohm's Law, the rest parts of I-V curve do not show the same behaviour. First, as shown in the figure 2, the starting points of oscillation region for the three samples are different. It is 3.5V, 4.5V, 5.5V for sample 1, sample 2 and sample 3, respectively. The 1V difference between the starting points can be a result of several possible reasons such as, for example, electrode polarization at anode or cathode, the resistance changes of electrolyte due to bubbles, and so on. In the following part of this paper, we can find that this difference between the starting points is larger in the BEP electrolyte than that in EP process. Another thing, which should be worth paying attention to, is the comparison between the sample I-V curves and the cavity I-V curve. We can find that the voltage corresponding to the maximum current in the cavity I-V curve is almost the same as that for sample 3 (see Fig.2). This is good news for us since the maximum current peak in the cavity I-V curve represents that furthest locations at the inner surface of the cavity from cathode enter into the oxide layer forming stage. For the oscillation region, it is a process for the forming of a compact solid film and then being partially removed by HF. Since the main component of the layer is considered to be Nb_2O_5 which is dielectric, the I-V curve decreases with the layer's increase until the thickness reaches a balance between oxide growth and removal. In this region, electrolyte Ohm's resistance or electric field analysis methods used in etching region is not suitable due to the forming of compact solid film and diffusion starts to play a main effect in the process. So, in this region, three samples' I-V curves decrease nearly in the same slope.

Then, after a little increase of current to obtain a balance point between the forming of compact solid film and the removal, three I-V curves of the button samples reach the polishing region. In the polishing region, there will be a little different between EP and BEP processes. As shown in figure 2 which is obtained in EP process, the three sample I-V curves in this region all show as nearly horizontal. We think this is because this region is mainly controlled by the mixture of concentration polarization and electrochemical polarization especially in EP process. The previous experimental data can prove this thought. So, the removal rate limited by the charge diffusion shows a voltage independent trend. However, we still can find those I-V curves have a slight upward inclination in this region. We think it is due to the fact that, apart from the diffusion mechanism, the charge transfer process may also be present. So, the slope in the higher electric field location will be steeper than the lower electric field location.

After the polishing region, there will be the gas evolution area. Usually, the work point should not be chosen in this area since the generation of oxygen bubbles

will leave pits on the surface. So, we will not discuss this part in details here.

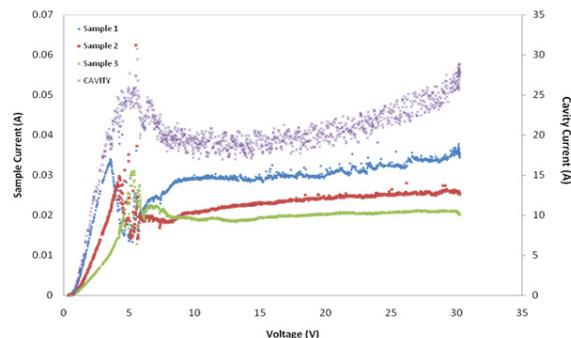


Figure 2: The I V curves in the EP experiment using the ball shape cathode.

I-V Characteristics of a Single Cell Cavity During BEP Process

Figure 3 shows the I-V curves during BEP process by using the demountable cavity. Similar to that in EP process, the typical regions in I-V curves appear for both button samples and the cavity. However, there also are many differences from those in EP especially in the etching and oscillation regions. As we can see, the difference in slopes between button samples 1 and 3 in the etching region is much greater than that in EP. Then the oscillation region of button sample 1 is also much larger than that in EP. This phenomenon should be caused by the properties of BEP electrolyte. Although, we will not use these two regions in electropolishing process, it will affect the identification of the working point. For example, if the etching region takes too large voltage range, perhaps we cannot find the suitable working point due to the ability of DC power supply. The study for it becomes one of critical points especially for BEP. We will discuss this in details in the next section of this paper.

The next region we will discuss is the polishing region. As shown in figure 3, in this region, the slope of I-V curve from button sample 1 is larger than that in EP. The slope of I-V curve from button sample 3 is relatively flat. However, the whole I-V curve from cavity shows an obvious slope. This should be from the contribution of the area near the iris. Thinking that one of greatest difference between iris and equator is the different electric field. We think that in BEP process, the charge transfer process might be more serious than that in the EP process. Thus, when the electric fields of sample are different at different locations on the cavity inner surface, the charge transfer rate can be different. This will be discussed in the following from the viewpoint of electric field distribution:.

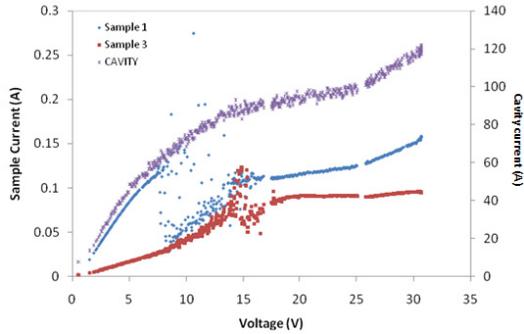


Figure 3: The I-V curves in the BEP experiment employing the ball shape cathode.

Electric Distribution from Small Sample Experiments

As referred above, we attribute the difference of I-V curve between button samples 1 and 3 to the difference in the distance from the samples to cathode. In this part, we will verify whether this thought is correct, and then try to find a method to analysis this problem in the cavity.

The experiments are carried out with two different distances of 3cm and 18cm with EP and BEP processes, respectively. Results are shown in figure 4. As we see, for the EP process, the results from the two different distances are not so much different. However, for the BEP process, a clear difference is found between the far and close distances. This result confirmed the effect of distance between anode and cathode. However, for a real cavity the distances between the cavity and cathode from one point to another are continuous. We cannot separate the surface to several areas which have the same distance from cathode as that in the sample experiments for analyses. So, the viewpoint of electric distribution is proposed, and the work for confirming the validity of this viewpoint is carried out with the further analyses for this series of small sample experiments.

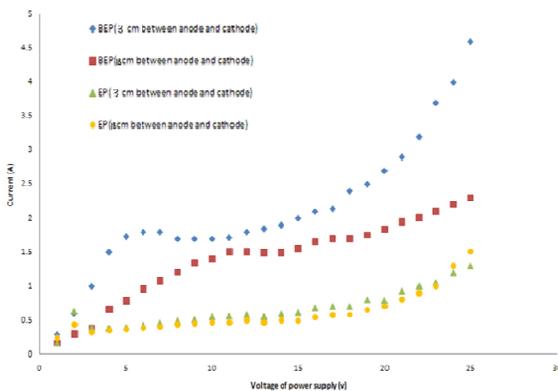


Figure 4: The I-V characteristics at two different distance in small sample experiments.

As we know, in an electropolishing system the voltage provided by power supply can be sorted into three parts according to the place it is dropped: anode, cathode and electrolyte, as shown in figure 5. The first two parts belong to the electrode polarization potential. Usually, the forming of polarization potential is complex and related to the properties of electrodes and the electrolyte. However, the measurement of the potential drops due to anode and cathode is no hard by using a reference electrode. Figure 6 is the potential drop on the anode surface measured by using reference electrode of Ag/AgCl. With the same method, the cathode polarization potential can also be easily measured.

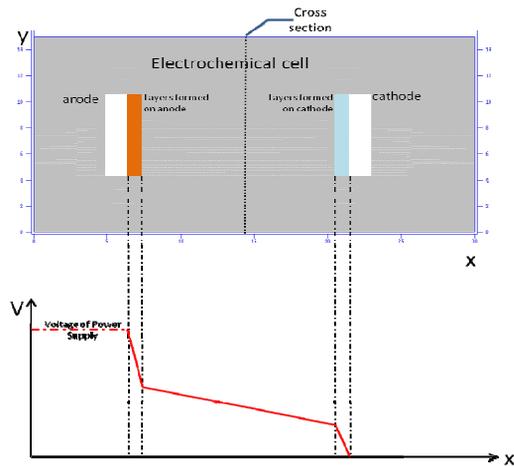


Figure 5: Schematic of small sample experiments.

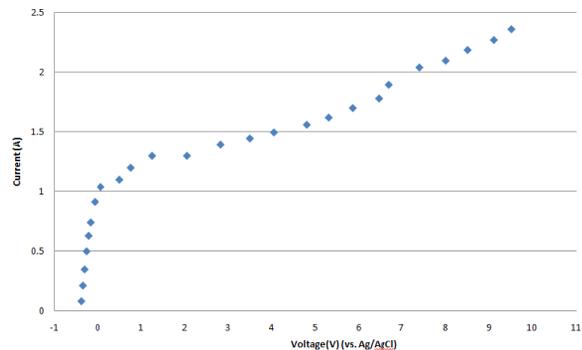


Figure 6: Anode polarization characteristic of Nb in BEP process with reference electrode Ag/AgCl.

Then the potential dropped on electrolyte can be easily obtained by using the voltage provided by power supply minus the voltages dropped on the two electrodes. Since distribution of electric field in the electrolyte is determined by Laplace's equation:

$$\nabla^2 \phi = 0$$

it can be obtained by numerical simulation via the program of Poission-Superfish as shown in figure 7.

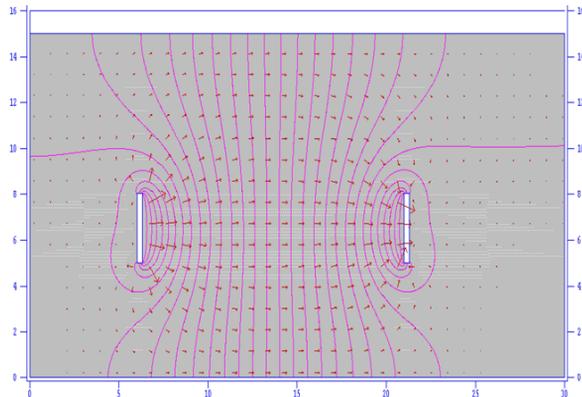


Figure 7: Simulation result of electric field distribution by Poission-Superfish in the small sample experiments.

To further prove the validity of this method, we have calculated the electrical conductivity at different voltages of the power supply. Since the current greatly varies with the voltage, if this method does not work, the result of electrical conductivity obtained at each voltage should be different. The detailed calculation process is done as follows: according to Ohm's law in electrolyte:

$$\vec{j} = \sigma \vec{E}$$

The current at the cross sections (see figure 5) can be calculated in the following way:

$$I = \iint \vec{j} \cdot d\vec{s} = \sigma z \int Exdy$$

So, we can obtain the electrical conductivity:

$$\sigma = \frac{I}{z \int Exdy}$$

\vec{j} is current density; \vec{E} is electric field; I is the current in the experiment; z is the height of electrolyte; x is the width of electrolyte; $\int Exdx$ can be obtained from numerical simulation.

The experiment is carried out in the range from 1V to 25V that is provided by the power supply. Figure 8 is the measured ratios of the potential drops in the electrodes and electrolyte relative to the whole voltage provided by the power supply. We can see from them, the voltage drops in the three parts have great changes with the increase of the voltage provided by the power supply. Figure 9 is the plots of the current and the calculated electrical conductivity as a function of voltage. We can see, although the current varies greatly at different potential, the calculated electrical conductivity is nearly the same. The standard deviation is only 0.0019. This

result indicates the validity of our analyses from the viewpoint of electric field distribution.

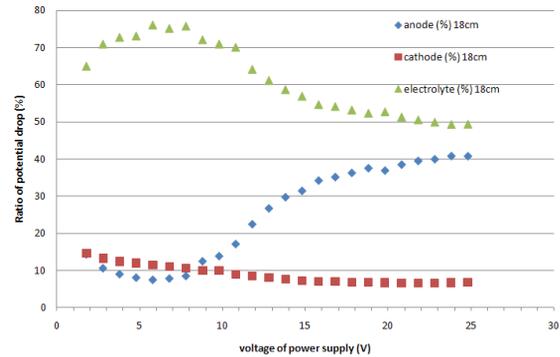


Figure 8: Ratios of potential drops for anode, cathode and electrolyte in BEP process at a distance of 18cm between anode and cathode.

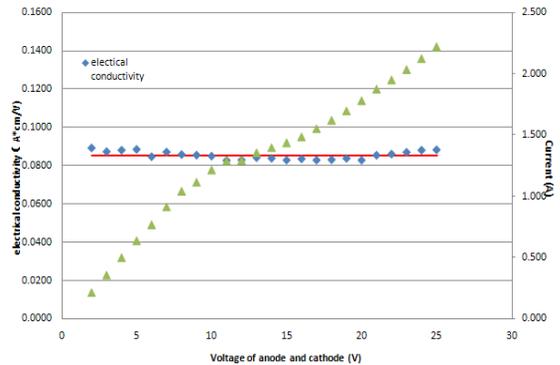


Figure 9: Plots of calculated current and electrical conductivity as a function of voltage.

CONCLUSION

The anode polarization characteristics for a single cell Nb cavity and at different locations on the inner surface of the cavity are studied by using a home-made demountable cavity. The experimental results indicate that there is an obvious difference at different locations especially for the etching region during BEP process. Similar tendency also appeared in EP although the difference there is not as great as that in BEP. Etching region will not play a role in the electropolishing process. However, it is critical for finding a suitable working point. The subsequent study of etching region in sample experiments shows that we can use the viewpoint of electric field distribution for this study. By using the measured I-V curves in the simulation employing Poission-Superfish, we can obtain identical electrical conductivities at different voltages in the sample experiments. It proves the validity of our field distribution viewpoint. Therefore knowing electric field distribution can be very helpful for real BEP or EP treatments on Nb single cell SRF cavities.

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