

## UPDATE ON THE R&D OF VERTICAL BUFFERED ELECTROPOLISHING ON NIOBIUM SAMPLES AND SRF SINGLE CELL CAVITIES\*

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### Abstract

Electropolishing (EP) has become a popular choice as the final step of the surface removal process during the fabrication of Nb superconducting radio frequency (SRF) cavities. One of the major reasons for the choice is that Nb SRF cavities treated by EP tend to have a better chance to reach an accelerating gradient of 30 MV/m or higher than those treated by buffered chemical polishing (BCP). This advantage of EP over BCP can at least be partially attributed to the smoother Nb surfaces that EP can produce. Recently a Nb surface removal technique called buffered electropolishing (BEP) was developed at JLab. The root mean square of the Nb surfaces treated by BEP can be as smooth as 20nm over a surface area of 200X200  $\mu\text{m}^2$ . In this contribution, R&D efforts of vertical BEP on Nb small flat samples and SRF single cell cavities since the last SRF conference in 2009 will be updated. It is shown that under a suitable condition, BEP can have a Nb removal rate as high as 10  $\mu\text{m}/\text{min}$  that is more than 25 and 5 times quicker than those of EP and BCP(112) respectively. Possible mechanisms responsible for the high Nb removal rate are proposed. Clues on the optimization of vertical BEP and EP treatments on Nb SRF cavities from recent experimental results obtained on a Nb single cell demountable cavity and Nb single cell cavities will be discussed.

### INTRODUCTION

Buffered electropolishing (BEP) was firstly proposed in 2001 by scientists at JLab [1]. Although several vertical electropolishing systems were built starting in 2002 for both BEP and conventional electropolishing (EP) [2], the main research focus of BEP then was on Nb small and flat samples using a bench top experimental setup. In 2008, it was realized that research on small samples could never substitute the R&D work on Nb SRF cavities [3]. Then more serious work on Nb SRF single cell cavities was launched formally. A much improved vertical treatment system with better temperature stability together with a computer data acquisition system was constructed recently as reported in Ref.4. Due to the limited

manpower and funding, the R&D on BEP has been carried out in an on-off fashion over the last decade. So far it has been demonstrated that BEP can produce much smoother surface finish as compared with those treated by EP and buffered chemical polishing (BCP). The polishing rate of BEP can be as high as 10  $\mu\text{m}/\text{min}$ . Sulphur precipitation should be much less (if any) from BEP than that from EP, since most of  $\text{H}_2\text{SO}_4$  is replaced by  $\text{C}_3\text{H}_6\text{O}_3$  in the electrolyte. The highest accelerating gradients of Nb single cell cavities have reached 32 MV/m on a large grain cavity of ILC shape and 28.4 MV/m on a fine grain cavity of cebaf shape.

In this contribution, the R&D on BEP since the last SRF conference in Berlin is reviewed. Effort has been put on the systematic understanding and optimizing of the polishing parameters and treatment procedures of BEP on a demountable Nb single cell cavity and normal Nb single cell cavities employing the improved vertical polishing system as reported in Ref. 4. Several cavities of cebaf shape treated by BEP have reached the highest accelerating gradients ever achieved on these cavities in the neighborhood of 25 MV/m. Mechanism has been suggested to explain the high polishing rate of BEP [5,6]. A new cavity fabrication procedure based on BEP was proposed in a collaborative effort between JLab and Peking University (PKU).

### OPTIMIZATION OF CATHODE SHAPE

Early experiments had shown that cathode shape could affect the polishing results of Nb SRF cavities for both BEP and EP [1], whereas the effect on BEP was much more serious. With the help of a demountable cavity, a more systematic study on this topic has been carried out for both BEP and EP [7]. Cathodes of various shapes such as, for instance, straight bar, ellipsoid, ball, and various wheels have been employed in the experiments. It was demonstrated that preferential polishing on the inner surface of a cavity could be realized by changing the cathode shape. Surface areas that showed a large electric field in the stationary electrolyte condition normally had a higher Nb removal rates.

To better understand the experimental results, it is useful to see how the demountable cavity looks like. Figure 1 shows the demountable cavity. This demountable cavity

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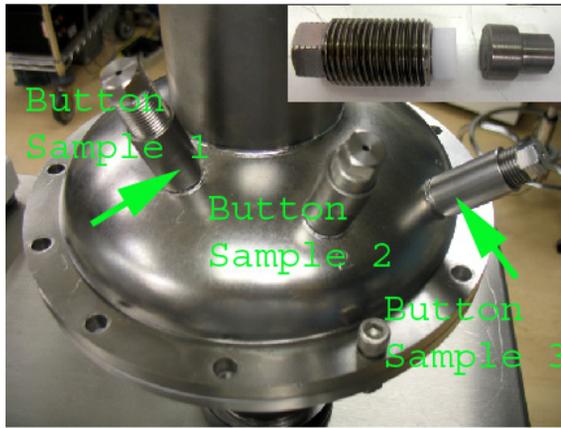


Figure 1: Photo of the demountable cavity fabricated at JLab. The arrows indicate the three button samples. The insert on the upper right corner shows the button sample assembly.

allows us to monitor the I-V curves of the whole cavity and the three button samples independently. The mass removal rates of the three button samples can be determined accurately by weighing the samples before and after polishing and the surfaces can be measured by various surface instruments.

Table 1 summarizes the results of Nb removal rates between Button Samples 1 and 3 for BEP and EP together with the results of electric field ratios between the two samples for various cathode shapes through the simulation via Poisson Superfish [7]. We can clearly see from the table that a more homogeneous polishing result can be obtained by optimizing the electric field distribution inside the cavity through the modification of the cathode shape given the conditions that temperature and electrolyte flow are kept constant for both BEP and EP. Based on this result, I suggest that conventional horizontal EP will have a more homogenous Nb mass removal of a Nb single cell cavity between iris and equator by using a shaped cathode as illustrated in Fig. 2. A more complicated cathode shape can also be designed since it is often desirable to remove more Nb at the equator because of the e-beam welding. Similarly, a shaped cathode should be used for EP on a multi-cell cavity. For the details of this study, please see Ref. 7.

Table 1: Removal rate ratios between equator and iris with different cathode shape in buffered and conventional EP. The electric field ratios were simulated employing Poisson Superfish (see Ref. 9 for details)

Cathode	Electric Field Ratio (Sample3/Sample1)	Ratio of Removal Rates for BEP (Sample3/Sample1)	Ratio of Removal Rates for EP (Sample3/Sample1)
Small Bar	0.13	0.12µm/0.57µm =0.21	0.43µm/0.85µm =0.51
Ball	0.16	2.07µm/4.28µm =0.48	0.24µm/0.48µm =0.50
Wheel	1.06	3.15µm/2.44µm =1.29	0.72µm/0.65µm =1.16
Large Bar	0.14	1.84µm/2.81µm =0.65	0.17µm/0.23µm =0.74

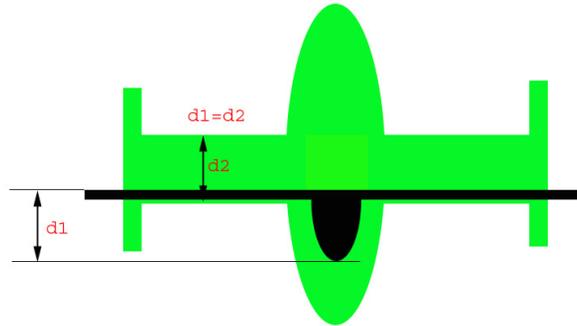
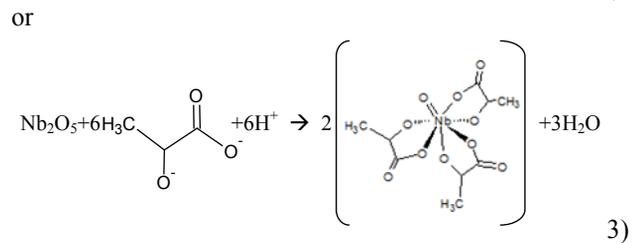
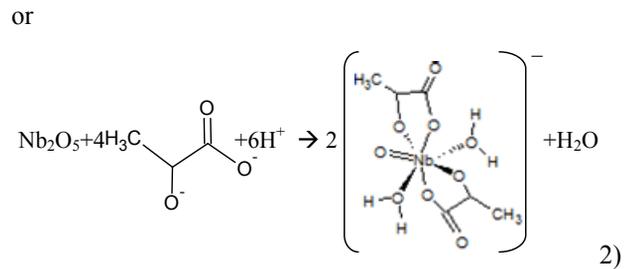
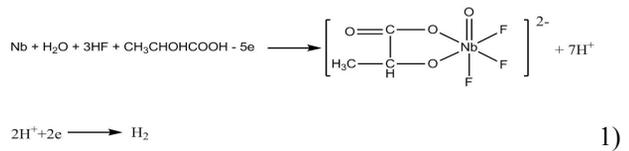


Figure 2: Schematic of a proposed cathode shape for a horizontal EP setup in order to have more homogeneous polishing on the inner surface of a Nb SRF single cell cavity based on the results of our R&D on BEP and EP. Similar idea can be applied to a multi-cell cavity.

### MECHANISM OF HIGH POLISHING RATE OF BEP

It has been demonstrated [8] that the Nb removal rate of BEP can be as high as 10 µm/min. Effort has been made to understand the much higher polishing rate of BEP as compared with that of EP. It is suggested [5,6] that apart from HF lactic acid also participates in the removal of Nb<sub>2</sub>O<sub>5</sub> in the following possible ways:



To test this idea, polishing was done with electrolyte consisting of H<sub>2</sub>O, H<sub>2</sub>SO<sub>4</sub>(96%), and C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>(85%) at a volume ratio of 4:5:11. A polishing rate of 6.8 nm/min was found [5]. This result seems to indicate that the contribution from the participation of lactic acid in

removing Nb is small, if any. This mechanism alone is not possible to explain the much enhanced removal rate of BEP.

### POLISHING ON NB DUMBBELLS

BEP experiments have clearly indicated that cathode shape has a dominant influence on the surface finish of a Nb SRF cavity, while less serious for EP (but cathode shape does matter). However, the size of cathode is always limited by the diameter of beam pipe of a Nb SRF cavity, leading to the difficulty in optimizing the cathode shape. It is a great advantage therefore to polishing Nb dumbbells first and then to e-beam-weld them together to form single cell or multi-cell cavities. This will make the life in SRF world much simpler since one does not need to worry about hydrogen bubbles and some invisible particles or surface defects on the polished surfaces. It is also possible to avoid polishing on multi-cell cavities.

To realize this dream, a specially designed Al cathode was fabricated at JLab according to the design from PKU as shown in Fig. 3. This cathode was used to do polishing on Nb dumbbells at PKU employing their vertical polishing system. The result of the first trial is shown in Fig. 4. A very shiny and mirror like surface finish was obtained. The next step is to weld dumbbells together and add beam pipes and flanges, followed either by a light BCP removal of  $5\ \mu\text{m}$  + high pressure water rinse



Figure 3: Photos of the shaped cathode fabricated at JLab according to the design from PKU. a) top view, b) side view.



Figure 4: Photos of Nb dumbbells polished by BEP via the shaped cathode of Fig. 3 a) before BEP treatment, b) after BEP treatment.

(HPWR) or just simply HPWR before being evacuated for RF tests.

### HYDROGEN BUBBLE MANAGEMENT

Hydrogen gas is an inevitable by-product of BEP and EP treatments on Nb. In addition to hydrogen intake, hydrogen bubble movement can create traces on the inner surface of a cavity especially when the polishing is done vertically. Therefore, it is important to find a way to minimize this hydrogen bubble effect. This study may not be very critical for vertical polishing on single cell cavities. However, it will be certainly indispensable for vertical polishing on multi-cell cavities. To study this effect, Teflon mesh of different sizes were used to wrap around ball cathode during BEP

In the early days, we used a Teflon mesh with grid size larger than  $1.5\ \text{mm}$  to wrap around our ball cathode, trying to create an obstacle for hydrogen bubbles to move towards Nb anode in a similar way as that used in the horizontal EP production facility of our lab. In reality, this did not help at all. Almost the same amount of bubble traces were observed on the upper half cell as those when the bare cathode was used. I-V curves of the two cases are almost identical. A typical example is shown in Fig. 5. The polishing plateau started to appear near  $34\ \text{V}$ . For the Teflon mesh size of  $100\ \mu\text{m}$ , bubbles traces would still be seen on the cavity surface. The I-V curve showed a significant change of the slope near  $37\ \text{V}$  (see Fig. 5). Although hydrogen bubbles could be blocked completely when the Teflon mesh size of  $0.45\ \mu\text{m}$  was used, there was not a clear observable significant change of slope on I-V curve (see Fig. 5) up to the maximum output of our power supply of  $40\ \text{V}$ . Polishing was done at  $35\ \text{V}$  but the surface after polishing looked dull and rough. In this case, the polishing plateau is expected to appear at a higher voltage than  $40\ \text{V}$ .

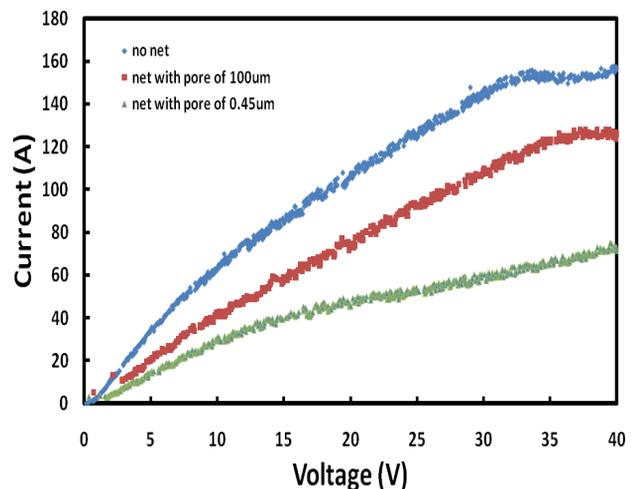


Figure 5: I-V curves of the ball shape Al cathode wrapped with bare and perforated Teflon sheets of difference sizes.

Further study to minimize the effect from the hydrogen bubbles through the change of the design of the cathode is underway and will be report in Ref.8.

### EFFECTS DUE TO CATHODE SURFACE AREA

Experiments have shown that cathode surface area has certain effects on polishing results for both BEP and EP [9]. This is due to the limitation from polishing dynamic of the electrochemical processes during BEP and EP. In both case, hydrogen gas is produced at the cathode as a result of positive H ions receiving electrons from it. When the cathode area is not large enough, the hydrogen gas produced at the cathode surface will block the charge exchange process from taking place there. This creates an unstable polishing situation.

This phenomenon was first noticed on BEP cavity treatment where bad surface finish was found when a cathode of a straight bar of a small diameter was used. To investigate this problem more carefully, detailed measurements of I-V curves were done on small flat samples of different ratios in cathode to anode surface areas ranging from 1% to 17% for BEP as shown in Fig. 6. The polishing plateaus show up only if the ratio is equal or larger than 10%. The same measurements were done for EP as well. It was found that at small ratios between cathode and anode surface areas, the I-V curves were highly unstable and oscillated too much to be able being recorded accurately.

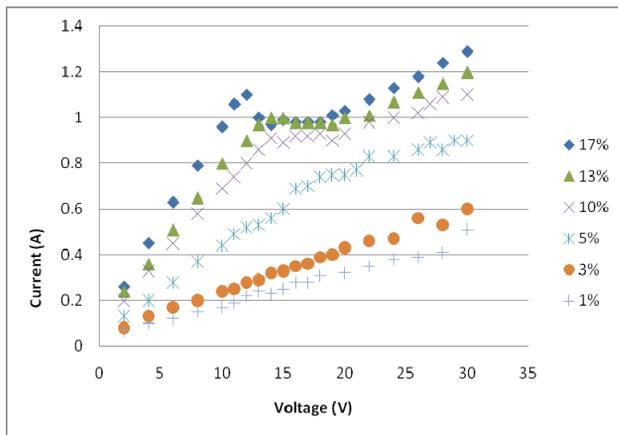


Figure 6: I-V curves of small sample experiments with different ratios between cathode and anode surface areas ranging from 1% to 17% for BEP.

### POLISHING ASYMMETRY BETWEEN UPPER AND LOWER HALF CELLS

This issue was recognized right from the initial stage of BEP development due to the fact that polishing was done vertically. Our preliminary solution was to flip the cavity orientation half way during polishing trying to average out the effect. However the issue was never a simple mathematic averaging. The surface finish of the upper cell is always different from that of the lower cell.

After introducing the demountable cavity (see Fig. 1), this issue could be visualized in a much better way. For BEP, it was always found that the upper cell had a brighter surface finish with the traces of hydrogen bubble movement while the lower cell appears to have more homogeneous surface polishing with almost no traces of hydrogen bubble movement. The lower cell was always less shinier than that of the upper cell as typically shown in Fig. 7 for BEP. The results of EP were the same. Recently a new demountable cavity was fabricated where button samples could be mounted on both upper and lower cells. This new demountable cavity will enable the study of the polishing asymmetry between the upper and lower cells to be done in a more quantitative and detailed way.



Figure 7: Photos of the upper and lower cells of the demountable cavity after treating by BEP using a wheel shape cathode.

### DEVELOPMENT OF POST TREATMENT PROCEDURE

As a new electrochemical treatment technique, BEP should have its own post treatment procedure. In the past few years, the post treatment of BEP has been overlooked. Simple treatment procedures of either BCP or EP have been adopted for treating on the cavities polished by BEP. Due to the presence of an organic acid in the BEP electrolyte, it is highly doubtful that either BCP or EP post treatment procedure is suitable for BEP processed cavities. The data of RF tests from those cavities have been scattering and some early on-set values near 15 MV/m or lower were observed.

Recently, a study of this problem was launched. A preliminary procedure consisting the following post treatment steps was found to be able to produce relatively better and more consistent RF test results has been developed [10]: A degreasing treatment after BEP, then ultrasonic cleaning, light BCP for 5µm, hydrogen degassing at 800°C heat treatment for 2 hours or 600°C for 10 hours, another light BCP for 5µm, HPR with JLab procedure for 40 minutes for a single cell BEP treated cavity and low-temperature baking at 120°C for 48 hours before RF test. After such a post treatment, a BEP treated.

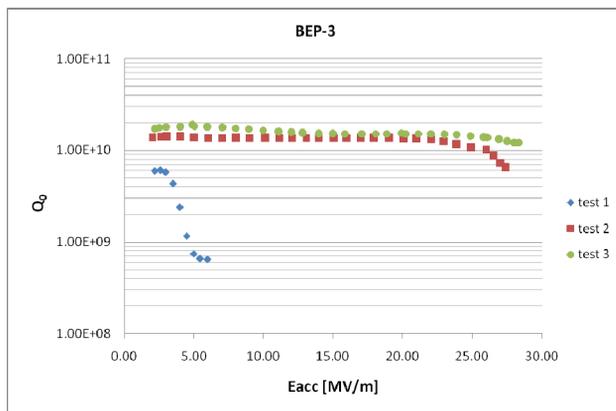


Figure 8: RF test results of BEP-3 at 2K before and after high pressure water rinsing as well as after the 120C baking.

Nb SRF single cell cavity of cebaf shape reached 28.4 MV/m, quench limited with a  $Q_0$  higher than  $1.2 \times 10^{10}$  at the quench point (see Fig. 8).

### SUMMARY

This contribution updates the R&D progresses on BEP since the last SRF conference in 2009. It was found that Nb SRF single cell cavities could be treated with a polishing rate as high as 10  $\mu\text{m}/\text{min}$  while the surface finish was still reasonably shiny and smooth. Several fundamental problems of vertical polishing issues such as, effect of cathode shape, mechanism of high polishing rate, hydrogen bubble management, effect of cathode surface area, and post treatment procedure have been under investigation. A suggestion on the shape of cathode was made for the conventional horizontal EP on a SRF single cell cavity in order to have more uniform polishing, based on the results of our R&D on BEP and EP. A study of a new SRF cavity fabrication procedure based on BEP polishing on Nb dumbbells employing a shaped Al cathode was initiated and the preliminary results were reported. A fine grain Nb SRF single cell cavity of cebaf shape treated by BEP has reached 28.4MV/m.

### ACKNOWLEDGMENT

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### REFERENCES

- [1] J. R. Delayen, et al., Proc. of 10<sup>th</sup> Workshop on SRF, Japan, (2001), P499.
- [2] A. T. Wu et al., Proc. of 11<sup>th</sup> Workshop on SRF, Germany, (2003), ThP13.
- [3] A. T. Wu, in the book *Neural Computation and Particle Accelerators: Research, Technology, and Application*, Nova Science Publishers Inc., New York, USA, (2010) Chapter 9.
- [4] A. T. Wu et al., Proc. of IPAC10, Kyoto, Japan, (2010), WEPEC080.
- [5] J. Beveridge and A. T. Wu, Report of CEU01, USA, (2010).
- [6] X. Y. Lu, Seminar at JLab, USA, (2009).
- [7] S. Jin et al., SRF2011 Conference, Chicago, IL, USA (2011).
- [8] A. T. Wu et al, to be published in Proc. of IPAC2011.
- [9] S. Jin et al., to be published.
- [10] S. Jin et al., to be published.