

# SYMMETRIC REMOVAL OF NIOBIUM SUPERCONDUCTING RF CAVITY IN VERTICAL ELECTROPOLISHING

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## Abstract

Vertical electropolishing (VEP) leads several advantages over horizontal EP in respect of easy operation and mechanism of an EP system resulting in lower cost. However, till yet VEP always resulted inhomogeneous removal of a niobium (Nb) cavity along its length and bubble traces especially on the top iris of a vertically set cavity. In this work we performed lab EP and VEP experiments in order to study and solve these two problems. A coupon cavity which contains 6 disk type Nb coupons positioned at beam pipes, irises and equator was vertically electropolished to optimize VEP parameters so as to get almost uniform removal of Nb and a smooth surface of the cavity without bubble traces. Our patented unique i-Ninja cathode having 4 wings was used with an optimized rotation speed to get homogeneous removal of Nb. The homogeneous removal and the surface without bubble traces might be result of a uniform thickness of a viscous layer on the surface of the cavity cell and no accumulation of hydrogen bubbles on the top iris surface. The surfaces of the coupons were studied in detail with surface analytical tools.

## INTRODUCTION

Electropolishing (EP) has been adopted as a promising method for final surface treatment of superconducting RF cavities. In order to carry out EP, horizontal EP (HEP) is used to get smooth surface and almost similar polishing rate for irises of a cavity cell. However EP rate for equator usually remains less than that on the irises. Vertical EP (VEP) has many advantages over HEP in respect of an EP setup. The VEP requires a simple setup compared to HEP setup since a HEP setup is built with complicated mechanism for cavity rotation and to make it vertical for drainage of acid. In spite of the advantages VEP has some drawbacks which includes inhomogeneous polishing of upper and lower half cells and bubble traces on the upper half-cell. Asymmetric removal might result in degradation of field flatness [1] while bubble traces enhance surface roughness of the upper half-cell of a cavity. To minimize the asymmetry a cavity is flipped to repeat the VEP as shown by other researcher. The flipping method is time consuming and makes cavity surface treatment expensive. In order to solve these issues we have developed Ninja cathodes, VEP setups [2-6] and

are further improving cathode and EP parameters. Here we report lab EP experiments to understand and solve the problem of asymmetric removal and apply similar technique in VEP of a cavity.

## EXPERIMENTS

### Lab EP Experiment

In order to understand the problems appeared in VEP and to solve them, three lab EP experiments, namely lab EP#1, 2 and 3, were performed. In Lab EP#1, four niobium (Nb) square samples in a size of 20×14 mm<sup>2</sup> were prepared for electropolishing. The samples were set horizontally at different positions near to a horizontally set aluminum (Al) cathode. Two Nb samples were set just above and below the cathode while both the samples were facing to the cathode. Other two samples were set aside to the cathode while both were facing downwards. A schematic and a photograph of a set of 4 samples in Fig. 1 show positions of Nb samples and Al cathode, and electrical connections for EP process.

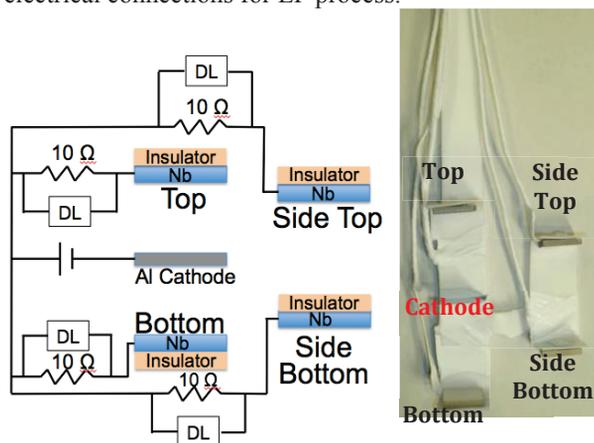


Figure 1: Schematic (left) for arrangement of Nb samples around an Al cathode and electrical connections to measure EP currents from individual sample. Photograph (right) of the samples and cathode.

As shown in the schematic, EP current can be measured for individual sample using data logger. The top/side top and bottom samples were set to simulate with top and bottom irises, respectively, of a cavity in VEP. The top and bottom samples were used to observe and compare effect of hydrogen (H<sub>2</sub>) bubbles generated from Al cathode during EP. The side top sample was set to observe indirect hitting of H<sub>2</sub> bubbles. The bottom side

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sample was set to observe effect of the gravity since H<sub>2</sub> bubbles generating from the cathode could not hit this Nb sample surface. An assembly of all the 4 samples and the cathode was set in an EP bath as shown in Fig. 2. EP bath temperature was maintained to be 19-22 °C.

Lab EP#2 was performed using other 3 Nb samples arranged in the same manner as in the lab EP#1 without the side bottom sample. This experiment was performed with a stirrer rotating at different speeds to understand effect of stirring on Nb surface. In the third experiment (lab EP#3), other 4 samples arranged as in the schematic were EPed at an optimized rotation speed of the stirrer.



Figure 2: EP bath for lab EP experiments. Inset: Stirrer used in lab EP#2 and #3.

### VEP Experiment

VEP of a 1-cell Nb coupon cavity was performed on 1-cell VEP setup [3] in order to optimize VEP parameters. The coupon cavity containing 6 Nb disk type coupons in a diameter of 8 mm at beam pipes, irises and equator positions. These coupons were set to the cavity with electrical isolation so as to measure EP currents from individual coupon as well as from the cavity. The cavity also contains view ports on the top and bottom irises to observe H<sub>2</sub> bubbles. The cavity has two more view ports on equator for light insertion. The coupons are taken out from the cavity for surface studies to know surface information of the different positions of the cavity. As a cathode our unique Ninja cathode, which is equipped with 4 wings was used. The wings are mainly made with non-metallic material but contain some metallic part as well. Hence Al rod and metallic parts of the wings were used as a cathode to perform VEP. The detail of the effect of metallic wings is reported in this conference [7]. VEP was carried out under the conditions listed in Table 1.

## RESULTS AND DISCUSSION

### Results of Lab EP Experiments

The lab EP#1 experiment was performed without stirrer for Nb samples arranged as shown in the schematic in Fig. 1. EP current densities were calculated by dividing

Table 1: Parameters for VEP of the Coupon Cavity with Ninja Cathode

Parameters	Values
H <sub>2</sub> SO <sub>4</sub> :HF	9:1
Electrolyte flow direction	Bottom to top
Electrolyte flow rate (l/min)	5
Cathode rotational speed (rpm)	50
Applied voltage (V)	~13-15
Target current density (mA/cm <sup>2</sup> )	~30
EP time (hours)	2
Target removal thickness (μm)	50
Maximum cavity surface temperature (°C)	~28

measured EP current by exposed area of the samples including the sides of the samples. EP current densities measured for these samples are plotted as a function of elapsed time in Fig. 3. In the beginning of the test data were not recorded by data logger. However the trend of current densities was the same in the beginning also. The highest current density was obtained from the top Nb sample while the side top sample showed the second highest current density. In comparison of the top and bottom samples, a lower current density was noticed for both the bottom samples. The current densities were measured to be almost similar for the bottom and side bottom samples.

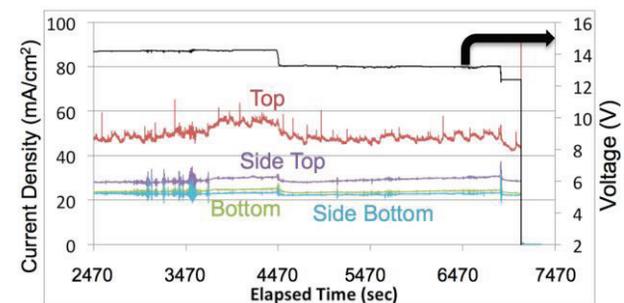


Figure 3: EP current densities for the Nb samples as a function of elapsed time of EP.

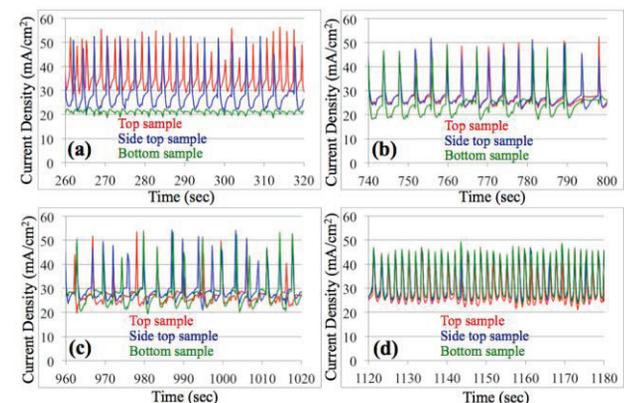


Figure 4: EP current densities for the top, bottom and side top samples at (a) 0 rpm, (b) 70 rpm, (c) 140 rpm and (d) 210 rpm.

In the lab EP#2 experiment a stirrer was set near to the samples and a rotation speed of stirrer was varied in order to know the effect of stirring. EP currents were measured for these samples at different stirring speeds of 0, 70, 140 and 210 rpm as shown in Fig. 3 (a)-(d). At 0 rpm EP current was the highest for the top sample similar as observed in the lab EP#1. When the rotation speed was increased, current densities of the bottom, side bottom and side top samples started to increase. At 210 rpm current densities for all the samples reached to very similar values (Fig. 4d). This experiment shows that stirring is important to obtain similar EP rate which can remain independent of positions of Nb samples.

The lab EP#3 experiment was performed at a constant and optimized rotational speed of 210 rpm for ~2 hours (for a short time the stirring speed was reduced to 140 rpm). In the beginning of the test rotational speed was set to 0 rpm in order to check reproducibility of the results as obtained in the lab EP#1. Figure 5 shows EP current density profiles for the 4 samples against elapsed time. The current densities for all the 4 samples at 0 rpm were similar to that in the lab EP#1. As already optimized in the lab EP#2, at the 210 rpm the current densities measured for the 4 samples were similar.

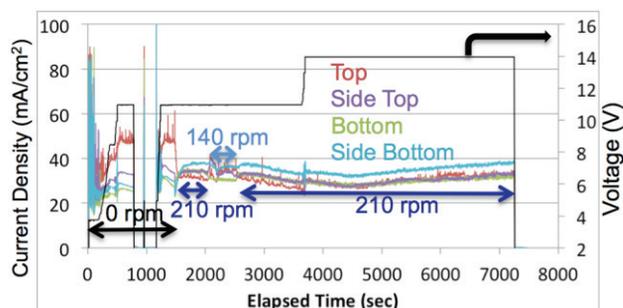


Figure 5: Current densities as a function of elapsed time of EP performed with a stirrer at 210 rpm.

### Removal Thickness and Surface Morphology of Lab EPed Samples

Removal thicknesses of the samples were calculated from their weight loss measured with a precision of 0.1 mg. The removal thicknesses are summarized in the Table 2. The comparison of removal thicknesses of the samples in the lab EP#1 shows that the removal thickness was the highest for the top sample and the second highest for the side top sample. The removal thicknesses are consistent with the current density results. The bottom and side bottom samples show almost similar removal thickness. In contrast to the lab EP#1 results, removal thicknesses of the samples after the lab EP#3 were found to be very similar.

Sample surfaces were observed with optical microscope and images of the samples treated in the lab EP#1 and #3 are shown in Figs. 6 and 7, respectively. In optical microscope images the side top sample surface was appeared to be rougher than the surfaces of other 3

samples. In lab EP#3 surfaces of all the samples were similar.

Table 2: Removal Thicknesses and Surface Roughness Data (Ra/Rz) for the Samples EPed in Lab EP#1 and #3

Sample	Lab EP#1		Lab EP#3	
	Removal Thickness (µm)	Ra/Rz (µm)	Removal Thickness (µm)	Ra/Rz (µm)
Top	77.8	0.25/1.3	47.6	0.44/2.2
Bottom	40.7	0.36/2.5	45.0	0.32/2.0
Side top	47.5	0.8/4.6	44.3	0.39/2.4
Side bottom	39.0	0.29/2.2	46.7	0.31/1.8

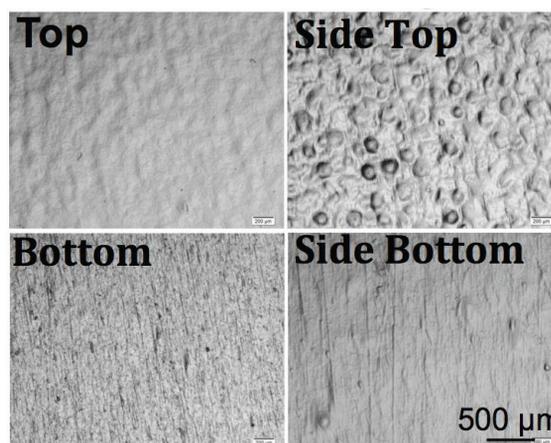


Figure 6: Optical microscope images of the 4 Nb samples EPed without stirrer.

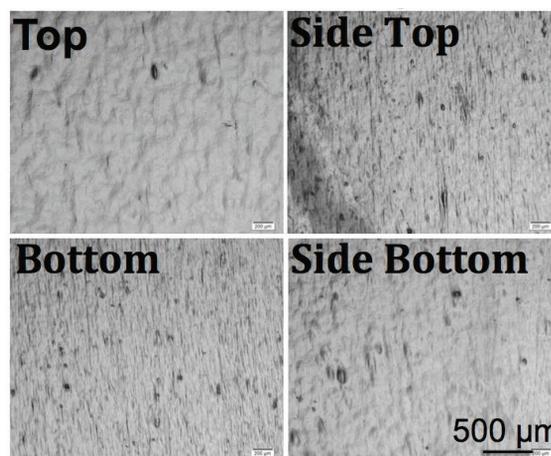


Figure 7: Optical microscope images of 4 Nb samples EPed with stirrer at 210 rpm.

To verify the result of optical microscope image surface roughness of the samples was measured with a mechanical surface profiler which can perform a line scanning on a surface to measure its roughness. The roughness data are shown in Table 2. The measured roughness for the top sample was found to be the lowest

whereas the side top sample was found with the highest roughness. In comparison of the bottom and the side bottom samples, roughness of the side bottom sample was less than that of bottom sample. However the difference in the roughness was not so large. In case of lab EP#3, similar surface roughness was measured for all the four samples. The results of removal thickness and surface roughness show that the stirrer speed of 210 rpm is very effective to obtain homogeneous removal and similar surface of the samples.

**Results of VEP Experiment**

The lab EP results reveals that the adequate rotation speed of a stirrer can result in uniform polishing remaining independent of sample positions. The top iris of a cavity in VEP is similar to the top/side top sample in the lab EP. In order to obtain the similar phenomenon in VEP as obtained in the lab EP at the high rotation speed, an adequate stirring is required during VEP. A VEP experiment was performed with a rotating Ninja cathode. Similar to the lab EP#2 experiment, rotation speed was optimized in our previous VEP which is not described here. A cathode rotation speed of 50 rpm was found to be adequate to get similar EP currents for the coupons set on the top and bottom irises. VEP was therefore carried out at a constant rotational speed of 50 rpm and using the VEP conditions described in Table 1.

EP current densities as a function of time for the iris and equator coupons are shown in Fig. 8. Current densities for the coupons in the cell (irises and equator) were very similar which show that EP should be uniform in the cell.

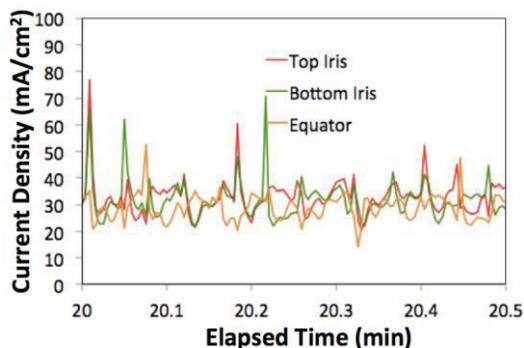


Figure 8: Current densities for the coupons at irises and equator.

**Removal Thickness of Coupons and Cavity**

In order to verify homogeneous Nb removal, removal thicknesses of the coupons and different positions of the cavity were measured. Average removal thicknesses of the cavity and coupons were calculated from the integrated EP current data whereas a local removal thickness at a particular position of the cavity was measured with an ultrasonic thickness gauge. An average

removal thickness of the cavity was calculated to be 47.7  $\mu\text{m}$ . The measured positions of the cavity with the gauge are illustrated in a schematic in Fig. 9 (a). These removal thicknesses were plotted against the length of the cavity as shown in Fig. 9 (b). The removal thicknesses of the coupons calculated from the integrated currents are also shown in the Fig. 9 (c). The removal thicknesses of the cell coupons are very similar with a difference of  $\pm 3 \mu\text{m}$ . The cavity removal thicknesses were found with larger difference compared to the coupons. The removal thickness on the top iris was  $\sim 1.5$  times larger than the bottom iris. Usually a removal thickness of the upper half-cell remains  $\sim 3$  times larger than the lower half cell as observed in our previous study and also by other labs.

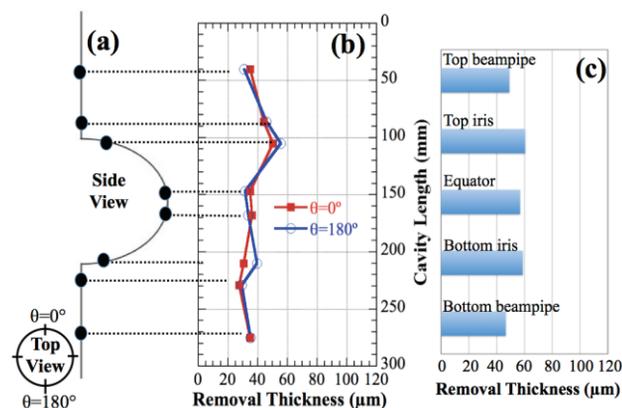


Figure 9: (a) Schematic showing thickness measurement positions on the cavity. (b) Removal thickness measured with an ultrasonic thickness gauge. (c) Removal thicknesses of the coupons calculated from the integrated EP current.

**Surfaces of the Coupons**

Optical microscope images of these coupons are shown in Fig. 10. The surface roughness for the coupons was measured with the surface profiler and the roughness data are shown in the Table 3. The surface roughness results shows Ra and Rz for all the samples were less than 0.4 and 2  $\mu\text{m}$ . The roughness is lower enough from the desired roughness [8] to achieve good performance of the cavity. Bubble traces, which usually appear on the top iris, were not found after the VEP. This shows that the Ninja is very effective to get smooth and uniform surface in the cell.

Table 3: Roughness of the Coupons after VEP

Coupon Position	Roughness (Ra/Rz) ( $\mu\text{m}$ )
Top beam pipe	0.24/1.4
Top Iris	0.36/1.8
Equator	0.25/1.3
Bottom Iris	0.19/1.1
Bottom beam pipe	0.27/1.4

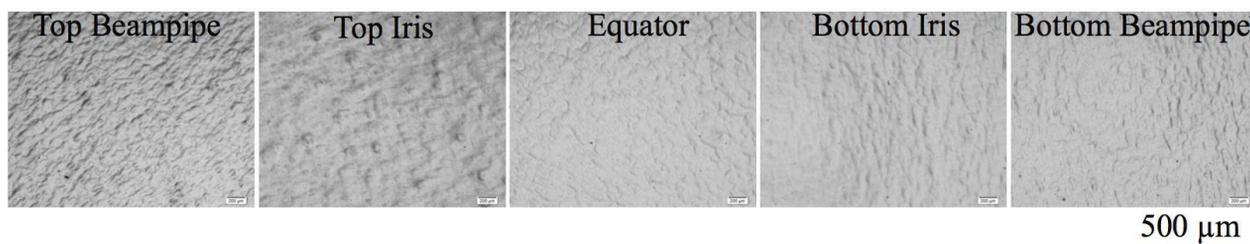


Figure 10: Optical microscope images of the coupons after VEP with the Ninja cathode rotating at a speed of 50 rpm.

### Discussion

In the lab EP#1 performed without stirrer, the highest removal of the top sample was probably due to direct attack of the H<sub>2</sub> bubbles releasing from the Al cathode. The H<sub>2</sub> bubbles might result in a thin viscous layer on Nb surface and enhanced EP rate for a higher removal thickness. The side top sample experienced hitting of comparatively less number of bubbles and hence the current density was found to be lower than that from the top sample. However it remained higher than both the bottom samples. Since both the bottom samples (facing upward and downward) were not attacked by H<sub>2</sub> bubbles generating from the cathode, the current comparison of these two samples can show effect of the gravity. The current for both the bottom samples were similar, which reveals that the effect of the gravity should be negligible in comparison to the effect of H<sub>2</sub> bubbles. However gravity may reduce surface roughness as seen in comparison of surfaces of both the samples. The smooth surface of the top sample was due to homogeneous accumulation of H<sub>2</sub> bubbles on the surface. The bubbles could not stay for a long time on the surface due to attack of other H<sub>2</sub> bubbles. In case of the side top sample the H<sub>2</sub> bubbles resided for a longer time to make thin viscous layer locally and hence create micro-rough surface. Similar rough surface of the top iris coupon was observed in case of VEP performed without stirring [3]. The rough surface was attributed to the longer residence time of bubbles which were observed on the top iris view port [3].

The stirrer at the high speed of 210 rpm might make uniform and laminar acid flow on Nb surface. The higher acid flow rate due to the high rotation speed did not let bubbles to accumulate on the top and side top samples. An adequate and uniform acid flow rate might generate almost uniform thickness of viscous layers on the samples' surfaces. As a result of the uniform viscous layer thickness and no accumulation of bubbles on the Nb surface, uniform polishing rate with uniform surface morphology was obtained.

Similar phenomenon was achieved in VEP with the Ninja cathode rotating at 50 rpm. At the 50 rpm bubbles displaced from the top iris surface as observed on the view port and acid flow might become uniform on Nb surface in the cavity cell. The uniformity is substantially improved in this VEP. The difference in the removal

thicknesses of coupons and cavity might be due to edge effect in which coupon edges at the high acid flow is polished at a higher EP rate compare to the flat surface.

### CONCLUSION

Three lab EP experiments were carried out for the horizontally set Nb samples around an Al cathode. The samples were arranged to simulate with top and bottom irises of a cavity in VEP. The lab EP#1 performed without stirrer resulted in inhomogeneous and rough surfaces specially the side top sample. In the lab EP#2 stirrer was used to agitate the acid and to clean the bubbles from the sample surfaces. A rotational speed of the stirrer was optimized to be 210 rpm to get similar currents from all the samples. In the lab EP#3 stirrer speed was kept to be 210 rpm to observe its effect on the surfaces of Nb samples. Removal thicknesses were found to be almost similar. To obtain homogeneous EP in VEP, Ninja cathode was used as cathode and stirrer. As a result of the Ninja rotation, symmetry in removal thickness was substantially improved and H<sub>2</sub> bubble traces were not found on the cavity surface. The Ninja cathode and VEP parameters solved the problem of serious asymmetry in removal thickness along the cavity length and bubble traces.

Further improvement in the cathode and VEP parameters is required to get perfect symmetry of Nb removal thickness in VEP of a cavity.

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