# PARAMETRIC STUDY FOR UNIFORM ELECTROPOLISHING OF 1.3 GHz NINE-CELL NIOBIUM CAVITY WITH DUAL FLOW MECHANISM

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#### Abstract

The interior surface of a niobium superconducting RF (SRF) accelerating cavity is treated with either horizontal electropolishing (HEP) or vertical electropolishing (VEP) methods, where the cavity is set in the horizontal and vertical positions in the HEP and VEP, respectively. A major challenge in the VEP is non-uniform material removal and surface roughness owing to the accumulation of hydrogen gas bubbles, generated on the cathode surface in the EP process, on the cavity surface. The presence of bubbles on the surface might impair the viscous layer of the electrolyte and enhances EP rate locally. The bubble accumulation was significantly reduced by applying a novel method in which the electrolyte was flown separately in a unique cathode housing and Nb nine-cell cavity. The effect of this dual flow was observed and EP parameters were studied by monitoring EP currents of the coupons fixed at the different positions of a test nine-cell coupon cavity. The removal asymmetry was reduced with the dual flow process and other optimized parameters. A 1.3 GHz nine-cell SRF cavity processed under the same conditions showed a good SRF performance at a temperature of 2 K.

#### **INTRODUCTION**

Niobium (Nb) superconducting radio-frequency (SRF) cavities are utilized in particle accelerators. The cavities are operated under superconducting state at cryogenic temperature typically at 2 K. The performance (accelerating gradient E and quality factor  $Q_0$  of the cavity depends on its interior surface. The interior of the cavity should have a smooth, damaged free, and contaminant free surface to meet the desired SRF performance. The top 100-200 µm damaged layer is removed by chemical treatments. Electropolishing (EP) process is applied for the surface treatments of high gradient cavities as the EP process yields a smooth surface. EP of the cavities can be performed by either putting the cavity in horizontal or vertical positions. Based on the position of the cavities during the EP, the process is named as either horizontal EP (HEP) or vertical EP (VEP). The HEP process as developed at KEK [1] is chosen as a successful method worldwide. However, the demerit of the HEP is that it requires complex and expensive setup. Alternatively, VEP process requires a simple setup to conduct the EP of the cavity surface. However, the nonuniformity in the removal along the cavity is quite strong in the VEP and the surface is usually found with the traces of gas bubbles. In the previous six years, we have conducted R&D on the VEP [2-5]. We have demonstrated that the asymmetric removal, in which the cavity cell shows higher removal near the top iris compared to that on the bottom iris and equator positions, is the result of accumulation of hydrogen (H<sub>2</sub>) gas bubbles [2]. H<sub>2</sub> gas is generated on the cathode surface as a reduction reaction on the cathode. In VEP, gas bubbles cannot be removed efficiently from the narrow aperture on the iris and a small diameter of the beam pipe. The bubbles finally gathered on the upper half-cell of the cavity to enhance the EP rate [2].

In order to mitigate the effect of gas bubbles, different models of our patented Ninja cathode were applied to the single cell cavities [2-5]. The cathodes were effective to attain uniform removal in the cavity cell and a smooth surface of the entire cavity [3-5]. Moreover, the cavity showed a good SRF performance in a vertical test conducted at 2 K [5, 6]. The parameters optimized for VEP of the single cell cavity were found to be inadequate for the nine-cell cavity.

This paper shows parametric study for VEP of 1.3 GHz Nb nine-cell cavity. The paper also includes the SRF performance of nine-cell cavity after bulk removal with adequate parameters of VEP.

#### **EXPERIMENTAL SETUP**

A parametric study was conducted with a test cavity namely nine-cell coupon cavity [7] and a unique Ninja cathode. The photographs of the cavity and schematic of the cathode is shown in Fig. 1. The cavity is having totally nine Nb disk coupons (in a diameter of 8 mm) on the top iris, bottom iris, and equator positions in the top, center, and bottom cells of the cavity. These cells also equipped with viewports near the top and bottom irises. The cavity allows to measure coupon currents and observation inside the cavity from the viewports during the VEP. The ninja cathode used in this work has the same design as used for the single cell cavity [3,4]. The cathode has insulating blades for agitation of acid in the cavity cell and a cathode housing with meshed Teflon sheet to stop bubble movement from the cathode housing to the cavity.

VEP of the nine-cell cavity is conducted with a system, which has facilities for carrying out VEP under different parameters for R&D. The facility is equipped with a cavity stand to fix the cavity in the vertical position, pumps for acid and water flow, acid tank with a capacity of  $\sim$ 70 L. The cooling of the acid and cavity is done with a heat ex-

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changer in the tank and by applying water spray on the exterior of the cavity. The cathode can be rotated at a variable speed during the VEP.



Figure 1: (a) Schematic of the coupon cavity with positions of Nb coupons and viewports. The photographs, on the right, of the iris coupons with viewport and equator coupon. (b) Schematic of the Ninja cathode and arrangement in the cavity cell.

### **RESULTS AND DISCUSSION**

#### Effect of Acid Flow

The study on acid flow was conducted. A new technique for acid flow was developed for fast discharging of gas bubbles out of the cavity. In this technique, acid is flown in the cathode housing and cavity separately. Flow rates in the cathode housing and cavity can be varied. The effect of the dual flow was studied with the coupon cavity. The coupon currents were measured under different flow rates as given in Table 1. In all the measurements, the cavity temperature was kept to be ~15 °C.

The coupon currents for flow rate at 5 L/min in the cavity without any intentional flow in the cathode housing were recorded. The cathode rotation was set at 20 rpm. Figure 2 shows coupon current density for all the nine coupons as a function of elapsed time. The coupon currents were similar in the bottom and center cells. In the top cell, the top iris coupon current started increasing after 30–40 s after the turning the voltage ON. The increase in the current was even higher when the cathode is not rotated. The higher current represents a higher removal rate.

Table 1: Acid Flow Condition in the Cavity and Cathode Housing

Test No. (Acid	Flow Rate (L/min)	
flow type)	Cavity	Cathode Housing
1 (single flow)	5	No intentional flow
2 (dual flow)	5	5
3 (dual flow)	5	10
4 (dual flow)	5	20
5 (single flow)	15	No intentional flow



Figure 2: Coupon currents in the top (a), center (b), and bottom (c) cells of the nine-cell coupon cavity. The curves were recorded at an acid flow rate of 5 L/min in the cavity with no intentional flow in the cathode housing. Cathode rotation of 20 rpm and cavity temperature bleow 15 °C were set.

The dual flow was applied under different flow rates in the cathode housing while keeping the flow rate same to be 5 L/min in the cavity (test no. 2–4 in Table 1). The curves under these conditions are shown in Fig. 3. The flow rate of 10 L/min in the cathode housing and 5 L/min in the cavity were found to be adequate for attaining similar coupon currents in the top cell.



Figure 3: The coupon currents in the top cell for the acid flow rate of 5 L/min in the cavity and an acid flow rate of 5 L/min (a), 10 L/min (b), and 20 L/min (c) in the cathode housing. The cathode rotation of 20 rpm and cavity temperature of below 15  $^{\circ}$ C were set.

The adequate flow rates in the dual flow method make the total flow rate of 15 L/min. In order to clearly understand the effect of dual flow, the same flow rate of 15 L/min was applied in the cavity without intentional flow in the cathode housing. The cathode rotation was not applied in these tests so as to eliminate the impact of acid flow due to the cathode rotation. The coupon currents under the two conditions are compared in Fig. 4. The current for the top iris coupon was higher in the case when no intentional flow was applied in the cathode housing. The top iris coupon current was reduced when the dual flow was applied with the same total flow rate. The results show the apparent effect of the dual flow to maintain the similar current in the cell.



Figure 4: The coupon currents in the top cell at (a) an acid flow rate of 15 L/min in the cavity and (b) an acid flow rate of 5 L/min in the cavity and 10 L/min in the cathode housing. No cathode rotation was applied. A cavity temperature was set to be  $\sim$ 15 °C.



Figure 5: Viewport located near the top iris in the top cell in the case of (a) single flow at a flow rate in the cavity was 5 L/min and (b) dual flow with a flow rate of 5 L/min in the cavity and 10 L/min in the cathode housing. Low brightness in (a) is due to a huge amount of gas bubbles in the top cell.

The higher current on the top iris in the case of single flow with 5 L/min in the cavity was attributed to the accumulation of gas bubbles on the Nb surface [2]. At 5 L/min, the discharge of bubbles from the cathode housing was not efficient. Gas bubbles generating on the cathode finally accumulate in a huge amount in the upper part of the cathode housing. In this case, bubbles diffuse from the cathode housing to the cavity via the meshed Teflon cover. The photograph of the viewport on the top cell is shown in Fig. 5 (a). The low brightness of the viewport is due to a huge amount of gas bubbles in the cell. Bubbles present on the Nb surface might affect the diffusion layer lying on the Nb surface to make it thinner. A thinner diffusion layer may allow diffusion of a higher concentration of fluorine ions which are responsible for dissolution of Nb material. In the dual flow, the flow in the cathode housing forces generating bubbles to move faster inside the cathode housing and reduce bubble diffusion to the cavity. The effect was confirmed by observing the viewport (see Fig. 5 (b)). As a result of this, similar coupon currents were attained.

### Polarization Curves for Coupons and Cavity

Polarization curves (current density versus voltage curves) were measured for the coupons and cavity under the dual flow condition. A polarization curve shows etching, oscillation, diffusion-limited and gas evolution regions [2], where the EP should be performed in the diffusion-limiting condition. The polarization curves for the coupons and cavity are shown in Fig. 6. The cavity temperature of ~15 °C and cathode rotation of 20 rpm were maintained during the test. The EP plateau, which represents the removal under diffusion-limiting condition, apparently appears for all the coupons and cavity. A slight shift in the EP plateaus towards the higher voltage was observed for the equator coupons in the upper cells. A shift in the plateaus was also found when the cavity temperature, cathode rotation speed, and flow rate of the circulating acid are enhanced.

The higher shift for the equator in the top cell was attributed to the cathode screening by H<sub>2</sub> gas bubbles. The number density of bubbles was obviously higher in the upper part of the cathode. The bubbles screen the cathode surface to reduce electric field on the Nb surface. In order to form an intact diffusion layer on the Nb surface, a higher voltage is required. The shift due to a higher cathode rotation speed might be due to an impact of higher acid flow on the Nb surface. A higher acid flow might affect the formation of diffusion layer and hence a higher voltage is required to obtain an EP plateau. A higher temperature operation enhances the EP rate to raise the current. The bubble quantity increases proportionally with the increase in the current. A higher bubble quantity further enhances the cathode screening to reduce the electric field. In order to keep the EP plateaus within 20 V, a cavity temperature was preferred to be around 15 °C.



Figure 6: Polarization curves for the coupons in the top cell (a), center cell (b), and bottom cell (c) and for the cavity (d). Cathode rotation of 20 rpm and temperature of  $\sim$ 15 °C were set.

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# Effect of EP Time

Effect of an EP time on the coupon current was also monitored. The top iris coupon current in the bottom cell started increasing after 3 min of voltage-turn on. It was observed from the viewport in the bottom cell that the bubbles returned back to the cavity from the acid tank. In order to reduce the number of returning bubbles to the cavity, the voltage was applied with on-off periods. Both on and off periods were set to be 3 min.

# VEP of Nine-Cell Cavities

The VEP of two nine-cell cavities (TB9-TSB02 and TB9-TSB01) were performed. The cavity TB9-TSB02 was vertically electropolished (VEPed) under unoptimized conditions, which were similar as optimized for the single cell cavity. The cavity TB9-TSB01 was VEPed with optimized conditions based on the above parametric study. The conditions for both the VEP are listed in the Table 2.

The cavity TB9-TSB02 was VEPed for an average removal of 51  $\mu$ m at an average current density of ~22 mA/cm<sup>2</sup>. The cavity TB9-TSB01 was VEPed for 100  $\mu$ m removal at an average current density of ~15 mA/cm<sup>2</sup>. The removal rate for TB9-TSB02 and TB9-TSB01 were calculated to be 0.28 and 0.1  $\mu$ m/min, respectively. Total time including voltage-off period was considered for the calculation of the removal rate for the TB9-TSB01 cavity.

The equator surface in all the cells was observed with an inspection camera known as Kyoto camera [8]. The equator surfaces are shown in Fig. 7. In the case of the unoptimized parameters, the upper four cells were found to be rough. The roughness was the largest for the top cell. On the other hand, the optimized conditions yielded smooth surface in all the cells.

Table 2: VEP Conditions Applied to TB9-TSB02 and TB9-TSB01 Cavities

Conditions	TB9-TSB02	TB9-TSB01
Acid	$H_2SO_4 (98wt\%):HF (55wt\%) = 9:1$	
Acid flow	Single Flow	Dual Flow
Acid flow rate	5 L/min	5 L/min in cavity & 10 L/min in cathode housing
Voltage	15 V	18.5 V (with on-off cycles)
Cavity Temperature	22 °C (max)	~15 °C
Cathode rotation	20 rpm	20 rpm

Removal trends in these two VEP are also compared in Fig. 8. Strong asymmetry in removal was seen along the cavity length when the unoptimized conditions were applied. Removal asymmetry within the cell increased towards the top cell. The asymmetry was significantly reduced with the optimized VEP conditions. The results corroborate with the coupon current study performed under different flow conditions. The strong asymmetry in the cavity TB9-TSB02 can be explained by the higher current observed for the top iris coupon in the top cell at 5 L/min in the cavity with no intentional flow in the cathode housing.



 $12 \text{ mm} \times 9 \text{ mm}$ 

Figure 7: Images of the equator positions in the cavity cells. The images in the left represent the equator surface of the cavity TB9-TSB02 after VEP with unoptimized conditions. The images in the right represent the equator surface of the cavity TB9-TSB01 after VEP with optimized conditions. The cell-1 represents the top cell in VEP.



Figure 8: Removal trends in VEP of TB9-TSB02 with unoptimized conditions (a) and in VEP of TB9-TSB01 with optimized conditions. The vertical lines indicate average removal thickness.

#### RF Test of Cavity TB9-TSB01

The cavity TB9-TSB01 was tested at 2 K in a vertical cryostat after the following treatments. After the bulk removal of 100  $\mu$ m, the cavity went through annealing in vacuum furnace at 750 °C, light EP of 20  $\mu$ m, annealing in vacuum furnace at 750 °C, light EP of 10  $\mu$ m, and baking at 120 °C for 48 h. The  $Q_0(E)$  curve is shown in Fig. 9. The cavity reached the maximum *E* of 28 MV/m at  $Q_0$  of 6.7x10<sup>9</sup>. The curve is compared with the baseline  $Q_0(E)$  curve measured at 2 K after the standard HEP process. The result reveals that the vertically electropolished cavity showed the same SRF performance as that after the HEP.



Figure 9:  $Q_0(E)$  curves measured at 2 K in a vertical cryostat after HEP and VEP treatments followed by 120 °C baking.

#### **OUTLOOK AND FUTURE WORK**

A parametric study was conducted to find adequate VEP conditions for attaining uniform removal and a smooth surface of the 1.3 GHz Nb nine-cell SRF cavity. The optimized conditions were applied to the cavity TB9-TSB01 for an average bulk removal thickness of 100 µm. The effect of the applied parameters was confirmed with removal trend and surface morphology. The comparison was made with the removal trend and surface of the cavity TB9-TSB02 treated with VEP under unoptimized conditions. The removal asymmetry was significantly reduced with the optimized conditions. Moreover, the surface was found to be smooth in all the cells of the cavity TB9-TSB01. An SRF test was conducted for the cavity TB9-TSB01 at 2 K in a vertical cryostat and performance was compared with the baseline SRF performance attained after the HEP. The SRF performance was found to be the same after HEP and VEP treatments.

Further VEP experiments are planned to improve the removal rate and uniformity. A new larger tank will be used in order to stop the circulating bubbles. This could reduce the asymmetry as found within the cells. Additionally, a continuous voltage without off-period could be applied to enhance the removal rate to be double.

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