RECENT DEVELOPMENT IN VERTICAL ELECTROPOLISHING

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Abstract

Horizontal electropolishing (HEP) is being used for final surface treatment of niobium SRF cavities. However a HEP system is equipped with complicated mechanism that makes it expensive and enhances cost of surface treatment of cavities especially when mass production is considered. Vertical electropolishing (VEP) has been introduced by other labs and the research is being carried out to establish the VEP technique. The VEP system requires simple mechanism and has advantages over HEP setup. Positive results have been obtained from the VEPed cavities also as shown by other labs. However further improvement in a VEP setup, cathode and VEP parameters is required. Marui Galvanizing Co., Ltd in collaboration with KEK has been working for development of VEP system, optimization of cathode and VEP parameters to obtain uniform Nb removal with a smooth surface of a cavity. Here we report our recent development of VEP system, unique Ninja cathode and parameter optimization with a 1-cell coupon cavity containing 6 Nb disk coupons at the beam pipes, irises and equator. The coupon surfaces were analyzed to obtain detail of the cavity surface.

INTRODUCTION

Electropolishing (EP) has been carrying out for several materials to obtain smooth surface. In accelerator surface of niobium (Nb) SRF cavities also treated with EP process to achieve a high electric field gradient with high Q_0 value [1]. Rough surface limits cavity performance due to presence of sharp tip like structure which might act as field emitters at a high field. Surface contaminations also degrade performance of Nb cavity as the contaminants might enhance surface resistance and might act as field emitters. In order to reduce roughness of inner surface of an Nb cavity, electropolishing (EP) is being carried out to remove Nb from inner surface in a depth of 100 microns. KEK perform bulk EP for 200 µm removal followed by fine EP for ~20 µm removal of Nb. An optimized EP parameters and adequate temperature can provide a smooth surface and very less amount of contaminants like sulfur and fluorine. Treating a cavity with EP can solve above-mentioned problems.

EP of an Nb cavity is either performed horizontally or vertically. Horizontal EPed cavities have shown consistently good performance and hence HEP is being used for surface treatment of the cavities worldwide. Vertical EP (VEP) has several advantages over horizontal (HEP) in respect of cost effective setup and easy operation [2].

Study on VEP is being carried out at Cornell, JLab, Saclay and other labs to optimize its parameters to establish the VEP method. In VEP polishing rate is usually found to be non-uniform along the length of the cavity. The top iris is strongly polished while the bottom iris shows the lowest polishing rate. A removal thickness on the top iris is usually found to be ~3 times higher than that on the bottom iris [3,4]. The asymmetry can degrade field flatness and therefore more efforts are require to tune the cavity after VEP [5]. Another issue with VEP is bubble traces on upper half-cell of a cavity. When bubbles move on Nb surface with acid flow from bottom to top, bubble leave their footprint on Nb surface. In order to minimize the longitudinal asymmetry, flipping of the cavity to repeat VEP is being used by other groups. In 1-cell cavity symmetry might be obtained by flipping the cavity while achieving symmetry in 9-cell cavity might not be easy even though the cavity is flipped to vertically electropolished twice. The flipping of a cavity upside down for repeating VEP makes VEP process time consuming and expensive. Therefore for industry point of view the flipping process cannot be adopted. Cavity performance was obtained in vertical test at Saclay for 1-cell cavity [4] and at Cornell for a 9-cell cavity [6]. A field gradient of ~35 MV/m was achieved with a Q_0 value of $\sim 10^{10}$ for the 1-cell cavity and the performance was similar to that obtained with HEP of the same cavity [4]. The 9-cell cavity after VEP at Cornell also showed good performance as required for ILC [6]. The good performances of the cavities encourage us to further optimize VEP parameters. We are optimizing VEP parameters and cathode shape in order to solve issues in VEP and to reduce cost of surface treatment of cavities [3,7-11]. We have joint collaborations with Saclay (Marui-KEK-Saclay) and with Cornell University (Marui-KEK-Cornell University). Cavity performance test is carried out at Saclay while our unique Ninja cathode will be tested at Cornell University.

In this paper we report effect of two types of recently developed Ninja cathodes on surface of a 1-cell Nb cavity.

RECENT DEVELOPMENT AT MARUI

VEP Setups

Two setups were constructed for VEP of 1-cell and 9-cell cavities. However a 9-cell system can also be used

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for VEP of a 1-cell cavity. Both systems were intentionally fabricated with PVC material in order to reduce cost of the system to be used for mass production in industries. However the VEP setup can be modified with PTFE or other materials which might be more resistant against EP acid.

1-Cell VEP Setup

Figure 1 shows photograph of the 1-cell VEP setup. The system is equipped with separate pumps and pipe lines for acid and water, an AC motor for rotation of our unique Ninja cathode, air-cooling system for cooling of outer surface of the cavity and an EP tank with heat sink system inside the tank. The acid and water can be flown from bottom to top or vice-versa. The air-cooling ducts having holes to blow air were set surrounding a 1-cell cavity for almost uniform cooling of the cavity during VEP.

Figure 1: VEP setup for 1-cell cavity.

9-Cell VEP Setup

The 9-cell setup is shown in Fig. 2. This system is also equipped with the acid pipeline, water line, an AC motor for cathode rotation, EP tank, a waste water tank and an air-cooling room for cavity. Efficiency of the air-cooling, recent improvement with water spray cooling and heat sync for EP tank are described in reference [7]. Acid flow, air-cooling, power supply, motor rotation speed etc. can be controlled with a control unit. The same control unit can be used for the 1-cell VEP setup as well.

Figure 2: VEP setup for 9-cell cavity.

Ninja Cathode

Ninja cathode has already been introduced in our previous work [3,7-12]. The first Ninja cathode was prepared with four Al metal wings to use the wings as equidistant cathode from the Nb surface in the cell and as a stirrer. The cathode was found effective in order to agitate EP acid and to make uniform distance from the cavity surface. A comparison of two VEPs performed with a conventional rod cathode and metal wings Ninja under the same conditions was shown elsewhere [3]. The comparison results revealed that the Ninja cathode was effective in order to get smooth surface of the cavity. The rod cathode resulted in rough equator surface. However both types of cathodes resulted in inhomogeneous removal of Nb in the VEPs. In order to obtain homogeneous removal and smooth surface of the cavity the Ninja cathode and VEP parameters were modified. Two Ninja cathodes were prepared with insulating wings and with partial metal wings which can be used as cathodes and a stirrer. Fig. 3 shows Ninja cathode with partial metal wings.

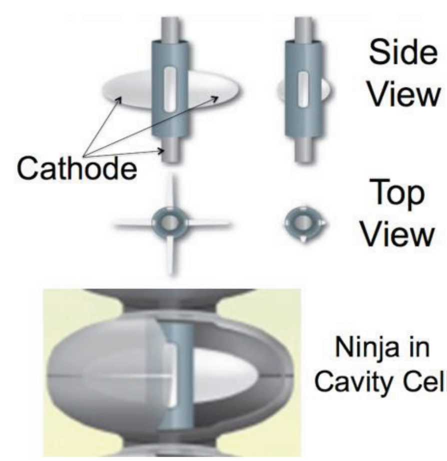

Figure 3: Schematic of Ninja cathode having partial metallic wings used as cathode.

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Coupon Cavity for Optimization of VEP Parameters

In order to optimize VEP parameters we are using a Nb coupon cavity which contains 6 Nb disk type coupons in a diameter of 8 mm. The coupons are set at the top and bottom beam pipes, top and bottom irises and two coupons at eqauator as shown in photograph of the cavity in Fig. 4. The cavity contains view ports as well to see inside during VEP. Effect of EP at different positions of the cavity is compared with EP current measured from individual coupons. To record the currents from individual coupons, the coupons are set to the cavity with electrical isolation. Moreover, EP flow, cathode rotation and bubbles attack can be observed from the view ports set on the top and bottom irises. Two other view ports on equator is also prepared for light insersion in the cavity.

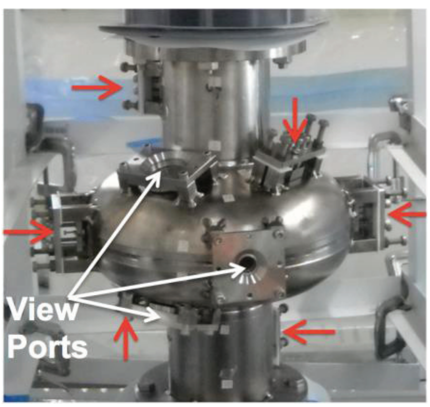

Figure 4: Nb coupon cavity with 6 Nb disk type coupons at beam pipes, irises and an equator shown by red arrows.

EXPERIMENTS

I-V tests were carried out with both the Ninja cathodes having insulating and partial metal wings. Two I-V tests were obtained for each cathode rotating at 0 and 50 rpm. EP currents were recorded for the individual coupons and the cavity at applied voltages ranging from 0 to 20 V. EP acid, acid flow direction, flow rate, and cavity surface temperature during I-V tests are listed in the Table 1.

VEP experiments for the coupon cavity were performed with both the cathodes to observe effect of the wings on coupons and cavity surfaces. Both the VEPs were performed under the conditions listed in the Table 1. The VEP with the insutlating wings was started with an applied voltage of ~19 V which was varied to keep cavity surface temperature lower than 30 °C. A final voltage was set to be ~13 V to carry out the VEP. For the VEP carried out with the partial metal wings, voltage was applied in a range of ~13-15 V. A rotational speed of the Ninja cathodes was kept to be 50 rpm for both the VEPs. Detail of the effect of rotational speeds in lab EP and VEP experiments is described in reference [12]. Air-cooling was applied to cool the outer surface of the cavity as shown in Fig. 1. The temperature of the cavity was maintained below 30 °C using the air-cooling and cooling of EP acid in an EP tank.

Table 1: VEP Conditions for the Ninja Cathodes of Insulating and Partial Metal Wings

Parameters	Insulating Wings	Partial Metal Wings
H_2SO_4:HF	9:1	9:1
Electrolyte flow direction	Bottom to top	Bottom to top
Electrolyte flow rate (l/min)	5	5
Cathode rotational speed (rpm)	50	50
Applied voltage (V)	~13	~13-15
Target current density (mA/cm^2)	25-30	30
EP time (hours)	2.5	2
Target removal thickness (µm)	50	50
Maximum cavity surface temperature (°C)	~25	~28

RESULTS

I-V Characteristics at Different Rotational Speeds of Ninja Cathodes

EP current versus applied voltage curves (I-V characteristic) usually shows 4 regions, namely etching, oscillation, polishing and gas evolution regions [13]. EP should be carried out in the polishing region to obtain smooth surface. Therefore adequate voltage or a range of voltage should be selected for EP. To understand effect of the two Ninja cathodes on different positions of the cavity, I-V curves were taken for individual coupons and cavity. The measured EP currents were divided by surface areas of the coupons and the cavity to calculate current density *J*. In the first two tests (test#1 and 2), the Ninja of insulating wings was rotated at 0 and 50 rpm, respectively. Current densities obtained at 0 rpm for the beam pipes, irises, two equator coupons and cavity were plotted as a function of the applied voltage as shown in Figs. 5 (a)-(d), respectively. The polishing regions for the top and bottom beam pipe coupons were obtained after a voltage of around 5 V (Fig. 5a). The polishing regions for the top iris, bottom iris and equator coupons were found shifted to the higher voltage side. The shift was found to be the highest for equator coupons. *J-V* curves for the coupons and cavity in the test#2 performed with a rotational speed of 50 rpm are illustrated in Figs. 5 (e)-(h). In comparison to 0 rpm, at the 50 rpm polishing region was further shifted for irises and especially for the equator coupons. The polishing regions for equator coupons seems not to be clearly appeared even at the maximum applied voltage of 20 V (see Fig. 5g).

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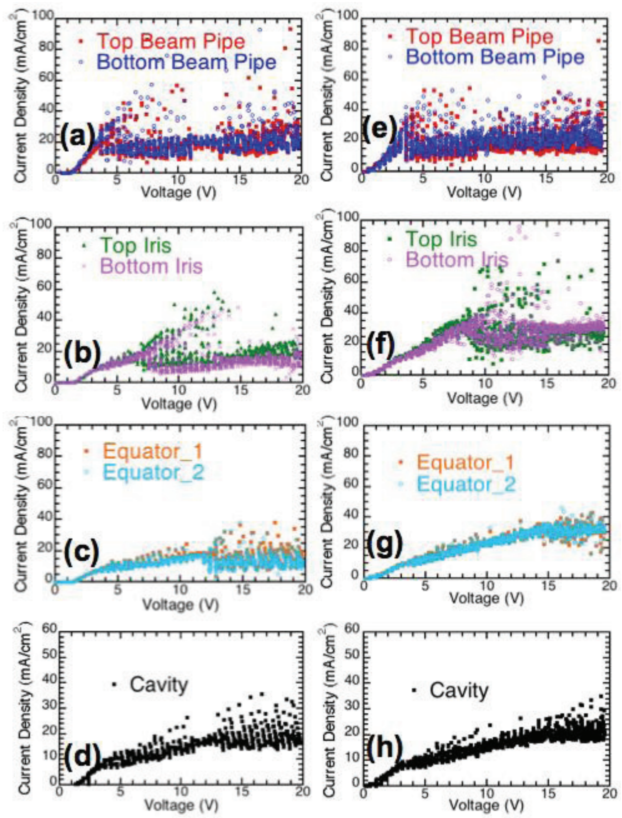

Figure 5: *J-V* curves obtained with Ninja cathode of insulating wings for beam pipes, iris, equator coupons and cavity at 0 rpm (a-d) and 50 rpm (e-h).

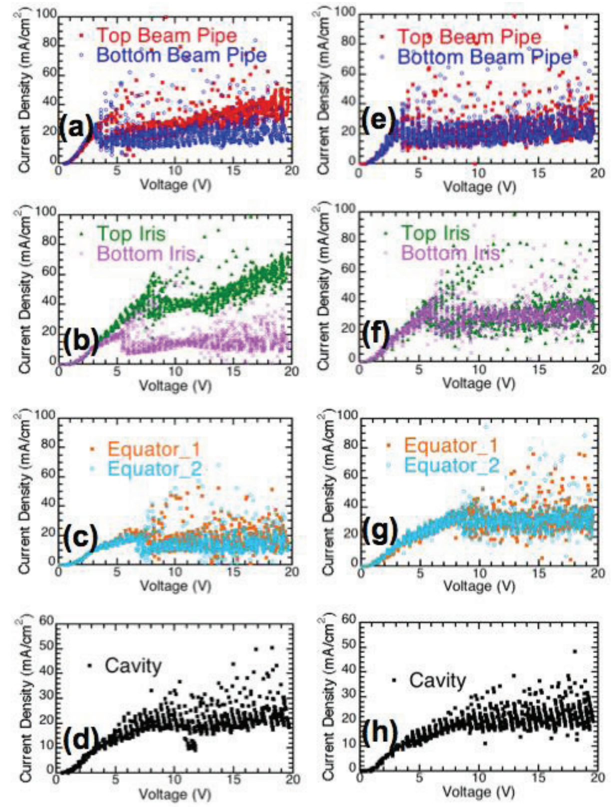

Figure 6: *J-V* curves obtained with the partial metal wings for beam pipes, iris, equator coupons and cavity at 0 rpm (a-d) and 50 rpm (e-h).

Other two more I-V tests (test#3 and 4) were performed with the Ninja of partial metal wings. In this case, rod and wings both acted as cathodes. The rotational speeds of the Ninja were set to be 0 and 50 rpm for the test#3 and 4, respectively. *J-V* curves are shown in Figs. 6 (a)-(d) for the test#3 and in Figs. 6 (e)-(h) for the test#4. At 0 rpm polishing region for the iris and equator positions can be obtained at a lower voltage than that required in case of the insulating wings. The top iris coupon current was increased and became the largest. Further shift in the polishing region was noticed for the iris and equator coupons at 50 rpm. The behaviour was similar to that obtained with insulating wings at 50 rpm. However the polishing region for the equator coupons could be obtained at a voltage of ≥11 V. At the rotational speed of 50 rpm, all the coupon current densities in the cell was found to be very similar. The results shows that the partial metal wings and the high rotational speed are very effective in order to obtain polishing region at a comparatively lower voltage and to get uniform polishing in the cell.

VEP Results

After the VEP experiments with both the cathodes, removal thicknesses of cavity were measured and surfaces of the coupons were studied as shown in the following sections.

Removal Thickness Comparison with Previous Result

Removal thicknesses of the cavity was measured using an ultrasonic thickness gauge at several positions as shown in a schematic in Fig. 7 (a). Figure 7 (b) shows removal thickness curve along the length of cavity after VEP performed with a conventional rod cathode as reported elsewhere [3]. The removal thickness on the top iris was measured to be around ~3 times higher than that on the bottom iris. Similar asymmetry after VEP has been noticed by other groups as well [4]. Removal thickness curves along the cavity length for the Ninja cathodes of insulating wings and partial metal wings are shown in Fig. 7(c) and (d), respectively. The longitudinal asymmtery was significantly reudced where the removal thickness on the top iris remained to be ~1.5 times larger than that on the bottom iris. The both new Ninja cathodes and recently modified rotational speed of Ninja cathode [12] reduced the asymmetry of Nb removal significantly.

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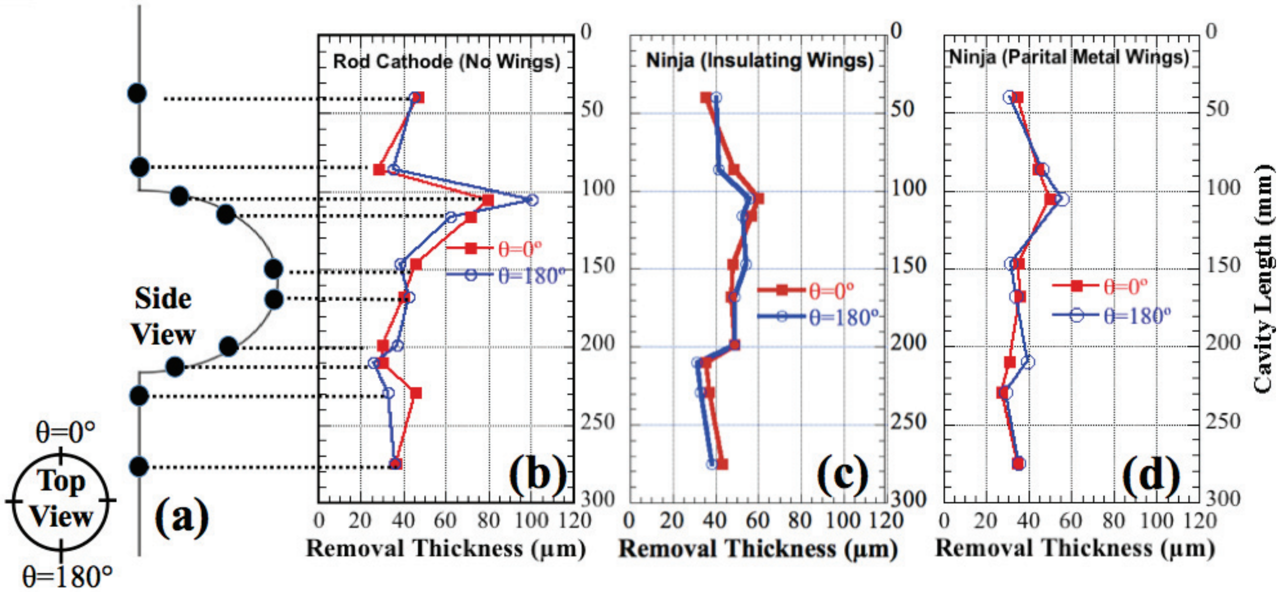

Figure 7: Comparison of removal thicknesses along the length of the cavity for VEPs performed with (b) a conventional rod cathode [3], (b) Ninja cathode of insulating wings and (c) Ninja cathode with partial metallic wings.

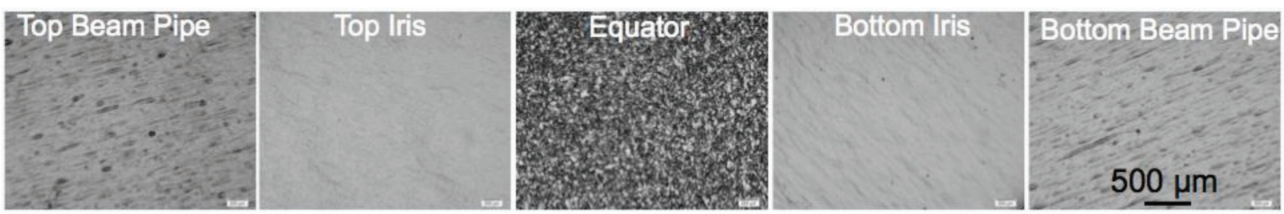

Figure 8: Optical microscope images of coupons after VEP with the Ninja cathode of insulating wings.

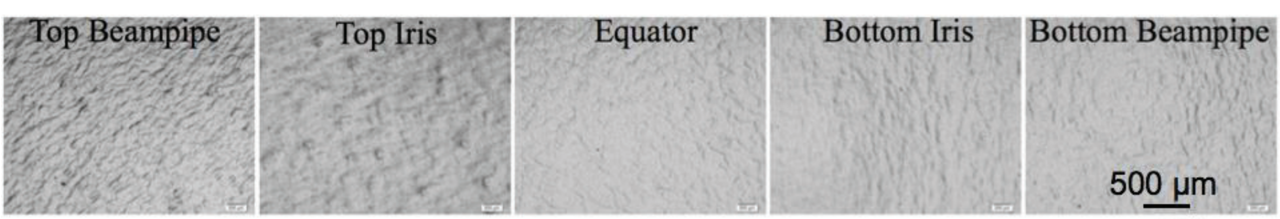

Figure 9: Optical microscope images of coupons after VEP with the Ninja cathode of partial metallic wings.

Surface Morphology of Coupons

Optical microscope images of the coupons are shown in Figs. 8 and 9 after both the VEPs. Surface roughness of the coupons was measured with a surface profile meter which can perform a line scanning for roughness measurements. Typical roughness Ra and Rz of the coupons before the VEPs was measured to be ~0.5 and 4 μm, respectively. The roughness results (Ra and Rz) after both the VEPs are summarized for all the coupons in Table 2. Ra and Rz of these coupons are compared in Fig. 10.

From the optical microscope images and roughness results, it is clear that equator surface was rough in case of the insulating wings. However the coupons surfaces on the irises and beam pipes were found to be very smooth. In case of the partial metal wings all the coupons including equator coupons were found to be very smooth. The partial metal wings were found effective not only to perform EP at the lower voltage but also to get a smooth surface of the entire cavity.

Table 2: Roughness of the Coupons before and after the VEPs with the Ninja and the Rod Cathodes

Sample Positions	Roughness Ra/Rz (µm) Insulating Wings	Partial Metal Wings
Top Beam Pipe	0.36/2.4	0.24/1.4
Top Iris	0.18/1.0	0.36/1.8
Equator	1.63/7.9	0.25/1.3
Bottom Iris	0.20/1.1	0.19/1.1
Bottom Beam Pipe	0.27/1.4	0.27/1.4

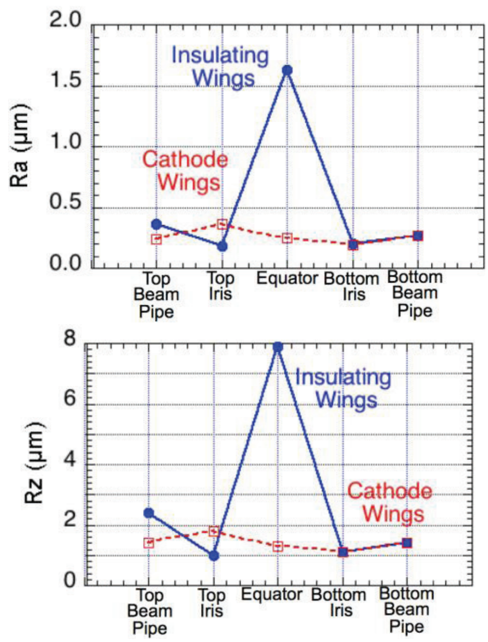

Figure 10: Comparison of roughness Ra (up) and Rz (down) of the coupons VEPed with insulating and partial metal wings.

Surface Analyses with XPS

After the VEP experiment with the Ninja of insulating wings, the coupons were disassembled from the cavity in a clean room/booth and loaded in a vacuum suitcase to transfer them in a X-ray photoelectron spectroscopy (XPS) analysis chamber at KEK. Surfaces of the coupons were analyzed with XPS to obtain atomic composition of elements and contaminants present on the surface. A large probing area of 700×300 μm^2 was selected for the analyses to get an averaged result of atomic concentration. Nb, O, C, S, F and Si elements were detected in wide XPS spectra for the top surfaces of the coupons. Narrow scans were conducted for these elements to calculate their atomic concentration. Sulfur (S), fluorine (F) and silicon (Si) were found on the surface as contaminants. Maximum atomic concentration of S, F and Si were calculated to be 1.4, 0.2 and 0.4%, respectively. The sources of S and F contaminants on the surface were H_2SO_4 and HF acid, respectively. Source of Si is unknown. Si might come from soil dust on floor since the VEP facility is not in a clean room.

DISCUSSION

In case of the insulating wings the *J-V* curves were shifted for the iris and further shifted for the equator. This shows that applied voltage required for EP depends on positions of the cavity. The equator positions which is far from the cathode might experience a lower electric field and therfore a higher voltage is required to reach in polishing region at equator position. The cathode surface is screened due to generation of H_2 bubbles in an EP process. The screening effect depends on a cathode area and quantity of H_2 bubbles which are proportional to an EP current. A small area of the cathode can easily covered with the bubbles which can reduce ion migration to the cathode. Due to the screening effect a higher voltage is required to enhance ion transportation to the cathode. In case of a small area of the cathode the distance between cathode and anode might become important. The beam pipe coupons were near to the cathode rod while the equator coupons are located at the most far distance from the rod. The large distance between the equator and the rod might reduce electric field on the equator. The Ninja cathode having partially metallic wings can enahnce an electric field on iris and equator. Moreover ions can transport to the additional cathode areas of the wings. In presence of the partial metal wings polishing region could be obtained at a comparatively lower voltage. At 50 rpm acid flow on Nb surface should be high and might be uniform. Due to a high and uniform acid flow, viscous layer thinckness on Nb surface might become uniform and thinner as well. As a result of a thin viscous layer, EP current increased and hence more H_2 bubbles are generated on the cathode area. The increase in H_2 bubbles further enhanced screening of the cathode and shifted *J-V* curves for iris and equator to a higher voltage side. Since wings are active only in the cell, *J-V* curves for beam pipe coupons were not affected at 50 rpm.

The longitudinal asymmetry was significantly minimized with both the cathodes rotating at 50 rpm. In our lab EP and VEP experiments it was turned out that accumulation of H_2 bubbles on upper half-cell enhances EP rate [3,13]. The higher EP rate on the upper half-cell resulted in asymmetry of Nb removal. Other groups reported that the non-uniform acid flow rate on Nb surface might be a cause of longitudinal asymmetry. An adequate rotational speed of the Ninja cathode can reduce bubble accumulation and also might result in uniform flow of acid on Nb surface.

The rough surface of the equator obtained in case of the insulator wings might be result of etching instead of polishing because equator remains in etching region even at the applied voltage of ~13 V (Fig. 5(g)). A smooth surface of equator might be obtained with the Ninja cathode of insulating wings when a highter voltage of ≥20 V is applied. However at such voltage iris and beam pipes might reach in gas evolution region which is also not desired. A smooth surface of equator and other positions

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was obtained with the partial metal wings because all the positions remained in the polishing region at the applied voltage of ~13-15 V. In case of the partial metal wings comparatively lower voltage is required for VEP. Therefore we can minimize applied power to keep a lower temperture of acid and cavity so as to perform VEP at a desired temperature.

CONCLUSION

VEP setups were constructed for VEP of 1-cell and 9-cell cavities. Two Ninja cathodes of insulating wings and partially cathodic wings were prepared and used in VEPs of a coupon cavity to compare their effect on cavity surface. Both the cathodes at a rotational speed of 50 rpm can minimize asymmetry along the length of cavity. However Ninja of insulating wing resulted in rough equator surface with Rz of 7.9 µm. *J-V* curves shows that different applied voltages are required to achieve polishing region at different positions of the cavity. With insulating wings, equator remained in etching region at an applied voltage of ~13 V during VEP. The EP performed in etching region resulted in rough equator surface. The partial metal wings might enhanced electric field on equator and ion transportation to the cathode. Polishing region for equator and irises was therefore shifted to a lower voltage in comparison to that with insulating wings. In case of partial wings VEP was performed in polishing region for all the positions of the cavity to get smooth surface of entire cavity. The Ninja cathode with partial metal wings could solve the problem of rough equator and minimize asymmetry of Nb removal along the length of the cavity.

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