VERTICAL ELECTROPOLISHING OF Nb COUPON CAVITY AND SURFACE STUDY OF THE COUPON SAMPLES

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Abstract

We have been carrying out vertical electropolishing of 1.3 GHz niobium (Nb) cavities for the ILC for 2 years. In this article we present results of investigation on vertical electropolishing (VEP) of a single cell Nb coupon cavity containing 6 Nb disk coupons located at beam pipes, irises and equator positions of the cavity. VEP were performed using our newly developed unique cathode called Ninja-cathode and a conventional rod cathode in order to observe and compare surface quality and the homogeneity of EP on the entire surface of the single cell cavity. Coupon currents for each coupon were measured to study EP phenomenon at different positions of the cavity. The surfaces of the coupons were analyzed with surface analytical tools like XPS.

INTRODUCTION

1.3 GHz niobium (Nb) superconducting RF cavities with a target field gradient of >35 MV/m and with a high Q value were adopted for the international linear collider (ILC). Rough surface and surface contamination remaining after the surface treatment degrade cavity performance. To achieve a good performance of cavities, surface treatments of the cavities play an important role. Electropolishing was considered as a promising technology for surface treatment of Nb superconducting RF cavities. Marui Galvanizing Co Ltd, Japan started vertical electropolishing (VEP) of Nb cavities in collaboration with KEK. We challenged to make a VEP system using PVC materials for the first time to aim drastic cost reduction. Moreover we are using our patented "Ninja" cathode of an especial shape having 4 retractable wings. The effect of the wings in agitation was demonstrated by flowing colored liquid having the same viscosity as EP solution in a transparent cavity [1]. In this article we show a comparison of results of two VEP experiments performed using the "Ninja" cathode and a conventional rod cathode.

VEP EXPERIMENTS

VEP experiments were carried out for a coupon cavity which was prepared with totally 6 holes on the beam pipes, irises and equator positions. The equator position contains two holes. 8 mm ϕ disk coupons which were electrically isolated from the cavity were fixed in these holes. The isolation allowed us to measure EP current from individual coupon. The cavity also contains 4 view ports on the top iris, bottom iris and equator. These view ports are used for in-situ observation and video recording especially for H_2 bubble attacks on the top and bottom irises. After the VEP the coupons are detached from the cavity and surfaces of these coupons are analyzed. These coupons provide us detail information of the inner surface at different positions of the cavity.

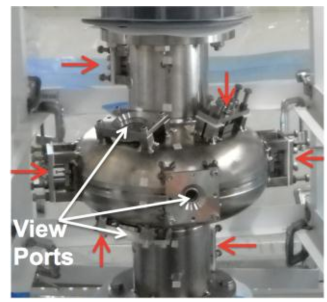

Figure 1: Nb coupon cavity with 6 Nb disk type coupons at beam pipes, irises and an equator shown by arrows.

Table 1: VEP Conditions for the Ninja and Rod Cathodes

Parameters	Ninja	Rod
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)	1	-
Applied voltage (V)	9-11	9
Target current density (mA/cm^2)	25	25-30
EP time (hours)	2.5	2.5
Cavity surface temperature (°C)	17.5-23	24.5-27

The coupon cavity was VEPed with the Ninja cathode and a rod cathode keeping the same parameters for both VEPs. All the VEP parameters are shown in Table 1. Electrolyte was circulated from bottom to top direction with a flow rate of ~5 l/min. During VEP the Ninja 4 wings cathode was rotated at 1 rpm for adequate agitation of the electrolyte. The flow rate and the cathode rotational speed were selected after several VEP experiments done with the Ninja cathode under different conditions. In order to cool the surface of the cavity during VEP we prepared a cooling system consisting of two hoses for passing the cool air from air coolers to the cavity surface. Each hose has equidistant holes to blow cool air uniformly on the beam pipes and irises for cooling. The cooling system maintained the cavity surface temperatures under 27 °C with variation of ~5 °C at different positions of the cavity. Both VEP experiments were performed for 2.5 hours for a target cavity removal thickness of 50 μm in average.

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RESULTS AND DISCUSSION

Coupon Current Profiles

A total current density and coupon current densities of the top iris, bottom iris and equator coupons are shown in Fig. 2. The top iris coupon current was always measured to be the highest whereas the bottom iris coupon current was the lowest. In one rotation of the Ninja cathode the top iris coupon current showed 4 peaks. These peaks were due to 4 wings which enhanced EP rate. The bottom iris and equator coupon currents were remaining very stable during the VEP with Ninja cathode. In case of the rod cathode many non-periodic current spikes were appeared in all the coupon currents.

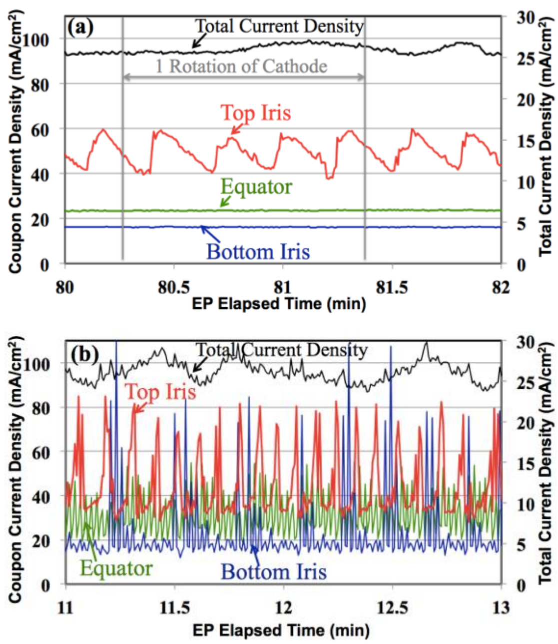

Figure 2: Coupon currents (a) Ninja and (b) rod cathodes.

H_2 Bubbles on the Top Iris

From the top iris view port as shown in the Fig. 3 it was noticed that the cathode wings regularly cleaned out the bubbles from the surface and agitate EP electrolyte. However the bubbles again appeared and remained on the surface until another wings passed the surface. In case of the rod cathode bubbles stayed on the view port surface for a long time and slowly moved up with EP solution flowing at the rate of 5 l/min.

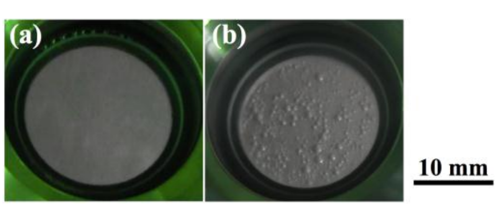

Figure 3: The top iris view port (a) just after passing a wing of the Ninja cathode and (b) rod cathode.

Surface Analyses of Coupons

After the VEP all the coupons were installed in a vacuum suitcase in a clean room. The suitcase is used for transportation of the coupons in vacuum and transfer of the coupons to X-ray photoelectron spectroscopy (XPS) analysis chamber in order to avoid the surface contamination due to dust particles from the atmosphere. The Nb coupons were analyzed by XPS intentionally with a large probing area of 700×300 μm² to get averaged results of existing contaminants on the cavity surface. The top surfaces of the coupons were mainly covered by Nb_2O_5 similar as reported by KEK [2] and other groups as well. Fluorine (F), sulfur (S) and silicon (Si) were also detected on the coupon surfaces after both the VEPs. The maximum atomic percentage of F and S were 0.5% and 0.1%, respectively. The maximum atomic percentage of Si was found up to 0.4% in the Ninja cathode VEP and up to 1.4% in the rod cathode VEP. Though in SEM and EDX analyses mainly carbonaceous and oxygen containing particles were observed, S and F were not detected by EDX. Si contamination might not be because of the VEP process itself but might be generated from some of the parts of the VEP setup or soil dust from floor of the factory since the VEP facility is not in a clean room. However the main source of Si and increase of Si in case of the rod cathode are still unknown.

In order to solve the Si contamination problem we are making our efforts to find out the source of Si contaminants. Simultaneously we are trying an alternative way to wash out Si from the cavity surface after VEP by rinsing the surface with electrolyte. For the test a Nb coupon containing >1% Si was rinsed with fresh EP electrolyte for 10 min in a lab. The Si was reduced to <0.4 % after the rinsing but could not be completely washed out. The rinsing method will be applied on the coupon cavity and the coupons will be analyzed with XPS to know the effect of this method on cavity surface.

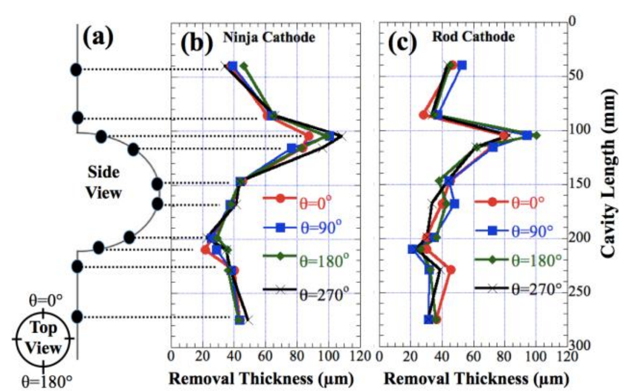

Figure 4: (a) Measuring positions for removal thickness. Measured removal thickness on these positions after VEP with (b) Ninja cathode and (c) rod cathode.

Removal Thickness of Cavity

Removal thickness at several particular positions of the cavity was measured by an ultrasonic thickness gauge. Measuring positions of the removal thickness were chosen at every 90° angle of the cavity circumference as shown by schematics of side and top views of the cavity

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in Fig. 4(a). The removal thicknesses at these positions are shown in Fig. 4(b) for the Ninja cathode VEP and in Fig. 4(c) for the rod cathode VEP. In both the VEPs the highest removal thickness was measured for the top iris where as the bottom iris was the position for the lowest removal thickness. The removal thickness at the top iris was around 5 times larger than that at bottom iris.

Surface Roughness of Coupons

An arithmetic roughness (Ra) and a peak-to-peak roughness (Rz) of all the coupons were measured before and after VEP with a mechanical surface profile meter (SPM). All the coupons were received from Tokyo Denkai, Japan and having similar initial surfaces. All the roughness data before and after the VEPs are summarized in the Table 2. In both VEPs Ra and Rz for the top iris coupon were always found to be the largest comparing to other 5 coupons. Roughness of the top iris and equator was increased with the rod cathode as shown in Fig. 5. Fig. 6 shows optical microscope images of a typical coupon surface before VEP and the top iris and equator coupons after both the VEP experiments. The images clearly reveal that the VEP with the rod cathode caused rougher surface at the top iris and equator.

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

Sample Positions	Roughness Ra/Rz (μm)			
	Ninja cathode		Rod cathode	
	Before	After	Before	After
Top Beam Pipe	0.53/4.2	0.28/1.8	0.57/3.9	0.31/1.8
Top Iris	0.51/3.3	0.50/2.3	0.43/3.4	2.98/13
Equator	0.43/3.2	0.22/1.3	0.50/3.4	0.94/5.3
Bottom Iris	0.45/3.2	0.28/2.2	0.46/3.7	0.26/1.8
Bottom Beam Pipe	0.39/2.7	0.27/1.6	0.52/3.7	0.24/1.6

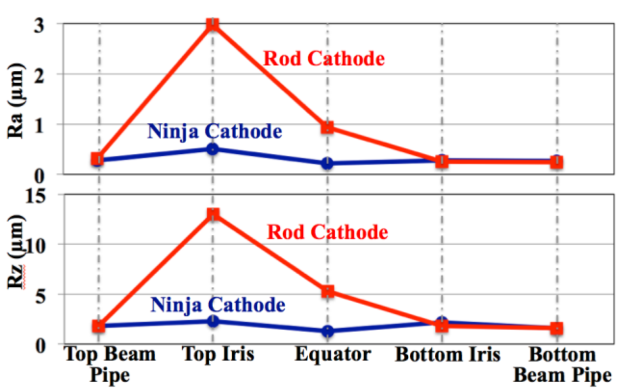

Figure 5: Roughness Ra and Rz of coupons after VEPs.

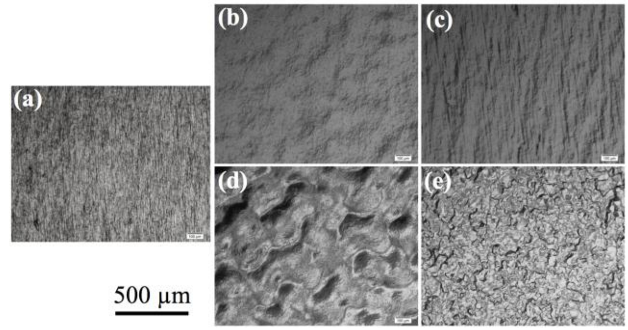

Figure 6: Optical microscope images. (a) Before VEP, (b) Top iris and (c) equator coupons after VEP with Ninja cathode, (d) Top iris and (e) equator coupons after VEP with rod cathode.

Discussion

The top iris compared to the bottom iris should have a thinner viscous layer (VL) due the gravity [3] and resident H_2 bubbles [4]. The thin VL might enhance an EP rate due to a lower resistance namely a higher current density shown in Fig. 2. This results in the higher removal thickness on the top iris comparing to the bottom iris [4].

In case of the rod cathode little agitation of electrolyte due to only its flow enhanced residence time of H_2 bubbles on the top iris as shown in Fig. 3(b). These sticky bubbles might locally attack and make micro-defects in the VL during their residence time. Electrical resistance might become further lower on the attacked area and hence the EP rate might become inhomogeneous at microscopic level. As a result of this the surface turned out to be rougher. The random current spikes in the coupon currents as shown in Fig. 2(b) were possibly due to these attacks. For the rod cathode the flow rate of 5 l/min might not be enough to move bubbles out quickly. However it was fond that the faster movement of bubbles caused traces on the upper half-cell as observed after our previous VEP done at 10 l/min in Marui and at 20 l/min in CEA-Saclay.

CONCLUSION

We successfully used a VEP system of PVC materials which is obviously cheaper than a PTFE system. A PVC made system can be used in industries for mass production and cost reduction of the cavity surface treatment. Results of two VEPs of the coupon cavity performed using the Ninja and rod cathodes were compared. With both cathodes removal thickness over the cavity was found inhomogeneous with ~5 times larger removal thickness at the top iris compared to the bottom iris. A smoother surface was obtained in case of the Ninja cathode. The Ninja cathode was found to be effective for agitation of electrolyte and to reduce residence time of H_2 bubbles on the surface. We need to improve the Ninja cathode and optimize other parameters to get not only a further smooth surface but also a homogeneous EP rate over the cavity surface.

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