STUDY ON VERTICAL ELECTRO-POLISHING BY CATHODE WITH VARIABLE-GEOMETRY WINGS

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Abstract

We have been studying on Vertical Electro-Polishing (VEP) of niobium (Nb) superconducting accelerator cavity for about one year with a view to the mass-production and cost-reduction of Electro-Polishing (EP) process. Marui Galvanizing Co. Ltd. has been in the EP business of various metals for long time and we have matured experience on EP processes. With being based on the experience, we thought that uniform electric-current on the surface of cavity and effective flow of electrolyte in the cavity are important factors. Moreover, we thought the most important effect is given if the cathode and the cavity surface (anode) are kept in a constant distance. Following these considerations, we invented VEP process by a cathode with variable-geometry wings. Using this cathode, we performed various tests of VEP with a Nb single-cell cavity as well as fluid circulation tests by a plastic mock-up of 9-cell cavity. In this article, we will report this unique VEP process, which might be applicable to the mass-production process of International Linear Collider (ILC).

INTRODUCTION

The Electro-Polishing (EP) is the method to remove and polish the inner-surface of niobium (Nb) Superconducting Radio-Frequency (SRF) cavity electro-chemically. The EP is thought to be the process to prepare the smooth inner-surface of SRF cavity and to achieve slightly higher accelerating gradient than Buffered Chemical Polishing (BCP) process where the inner-surface of cavity is removed chemically. In many laboratories, the removal thickness is more than 100 micrometer in the first EP process in order to remove the affected layer and roughness of surface caused by such fabrication processes as rolling, deep-drawing, welding and so on, and after a following firing process with 800 ^{0}C, a few tens micrometer would be removed in the second or final EP process to prepare smooth and clean inner-surface. Currently, the standard setup of EP process for elliptical 9-cell cavities is in horizontal posture of cavity, where we call it the Horizontal EP (HEP) setup hereafter. However, in the HEP process, the rotation of cavity is needed and the draining process of electrolyte is complicated, and then a large space is needed for the process, which might has some possibilities for the simplification. In such a situation, the Vertical EP (VEP) setup where the cavity is set up in vertical posture without rotation and the draining process is simpler is studied in laboratories around the world [1-5]. Because the simple VEP process is fundamental issue in the mass-production of cavities in the future SRF projects like International Linear Collider (ILC), we started the study on the VEP process in the collaboration of Marui Galvanizing Co. Ltd. and KEK about one year ago. We considered the most important points in the VEP process are the uniform electric-current densities on the inner-surface of cavity, in other words, the constant distance between the cathode and cavity surface, and the effective flow of electrolyte in the cavity. Then we invented a unique VEP process with variable-geometry wings. In this article, we report the concept, structure and advantage of the cathode with variable-geometry wings which we named i-cathode "Ninja"®, as well as the tests of VEP process with the i-cathode by a Nb single-cell cavity, and the tests to prove the effective flow of electrolyte with the i-cathode by a plastic mock-up of 9-cell cavity.

I-CATHODE "NINJA"®

The schematic view of the i-cathode "Ninja"® which we invented is shown in Fig. 1. The most important feature is that it has four wing-shaped aluminium electrodes on the aluminium rod at the center of cavity cell. The cathode has a retractable structure of flexible wings. When the cathode rod is inserted into or pulled out of the cavity before and after the EP process, the wings are retracted. When the cathode is once inserted into the cavity, the wings are unfolded with a simple action. This simple structure might contribute to realizing a short setting-time of EP process too.

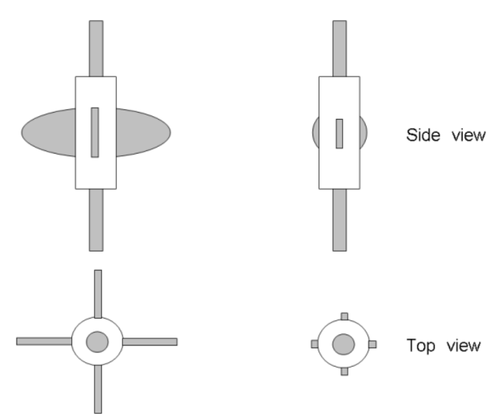

Figure 1: Schematic view of i-cathode "Ninja"® for a single-cell cavity. Left-hand side: unfolded status. Right-hand side: retracted/folded status.

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By making the i-cathode with wings rotated during the EP process, following two advantages are expected compared with the standard cathode structure of simple rod without wings. (1) The wing works as a baffle plate in the cell of cavity for the flow of electrolyte and this helps the uniform distribution of electrolyte in the cavity. (2) The wings make the distance between the cathode and the cavity surface (anode) shorter in the cell of cavity and realize the uniform distribution of electric field in the cavity. With these excellent features of i-cathode, the uniform removal of Nb material might be realized over the whole inner-surface of cavity.

UNIFORM FLOW OF ELECTROLYTE IN 9-CELL CAVITY

In order to find out the conditions for the uniform flow of electrolyte, we prepared plastic mock-ups of 9-cell cavity and i-cathode. Using the mock-ups, we performed experiments to see the distribution and flow of colored water in 9-cell cavity. Colored water was made with adding red food coloring to water and the similar viscosity to the 98% sulphuric acid was simulated by adding Carboxyl-Methyl-Cellulose (CMC) to colored water up to the concentration of 2.5 g/L. Following conditions are tried in the tests, (1) two directions of water flow: upward and downward, (2) two flow rates: 25 L/min. and 12 L/min., and (3) two conditions of i-cathode: unfolded wings with rotation and retracted/folded wings without rotation.

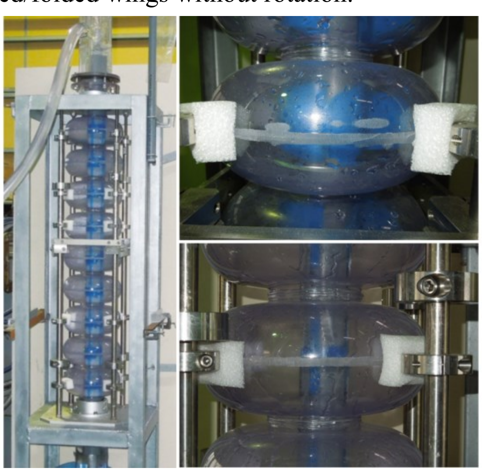

Figure 2: Left-hand side: pictures of setup with a plastic mock-up of 9-cell cavity to see the colored water flow. Right-hand side, upper: wings are unfolded and the cathode rod was rotated. Right-hand side, lower: wings are retracted.

In the results of experiments, when the flow rate is 25 L/min., the distribution and flow show no difference among (1) two directions of flows and (3) two conditions of i-cathodes. On the other hand, when the flow rate is 12 L/min. and only when the flow direction is upward, uniform distribution and flow were realized in the i-cathode condition of unfolded wings with rotation, but colored water flowed only near cathode in the i-cathode condition of retracted wings without rotation. The pictures of experiments for the flow rate of 12 L/min. are shown in Fig. 3. We considered that the wings might work as baffle plates much effectively for the colored water and realized uniform distribution of colored water in the i-cathode condition of unfolded wings with rotation in particular when the flow rate is low.

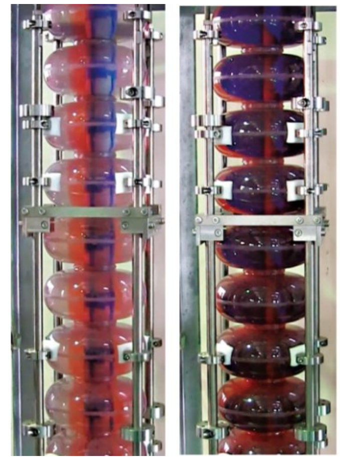

Figure 3: Pictures of experiments in the flow rate of 12 L/min. and in the direction of upward flow. Left-hand side: i-cathode condition of retracted wings without rotation. Colored water has dense distribution and flow near the cathode rod. Right-hand: i-cathode condition of unfolded wings with rotation. The colored water has uniform distribution and flow in the cavity.

VEP EXPERIMENTS OF NIOBIUM SINGLE-CELL CAVITY WITH I-CATHODE “NINJA”®

We performed Vertical EP (VEP) experiments of Nb single-cell cavity three times with i-cathode “Ninja”®.

First VEP Experiment

The picture and schematic view of setup for the first VEP experiment is shown in Fig. 4.

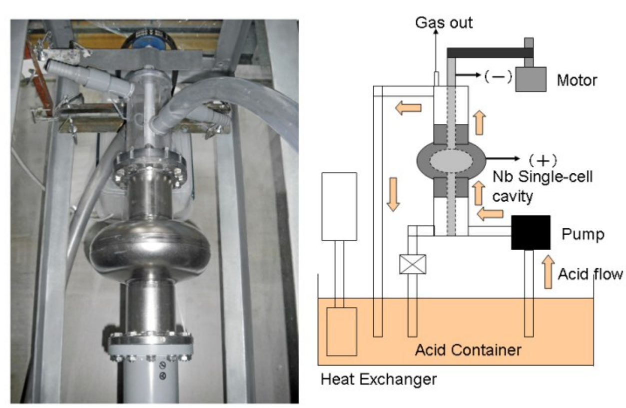

Figure 4: Picture (left-hand side) and schematic view (right-had side) of setup for the first VEP experiment.

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As seen in the schematic view in Fig. 4, cooling water was supplied from chiller to the heat exchanger in the EP electrolyte container. The sequence of VEP experiment was as follows. (1) The circulation of EP electrolyte started in the direction of upward flow. This is because we considered the bubbles in the EP electrolyte might be extracted more effectively and we had the better result in the previous mock-up experiment. (2) The i-cathode with unfolded wings started rotation. (3) The voltage was applied between the i-cathode and the cavity (anode). (4) if the temperature raised, the voltage was switched off for a while. Then (3) and (4) were repeated several times. The temperature was measured with six thermocouples, on the outer-surface of the cavity at the lower beam-pipe, near equator in lower cup, near equator at upper cup, upper beam-pipe as well as in the EP electrolyte at the upper outlet just above the upper beam-pipe and in the EP electrolyte container.

Table 1: Conditions of first VEP experiment

Items	Condition
Electrolyte composition	H_2SO_4(98%) : HF(55%) = 9:1 (V/V) and fresh electrolyte
Voltage	From 14to 9 V
EP duration	30 min. (continuous)
Flow direction	Upward (bottom to top)
Flow rate	5 L/min.
Cathode rotation speed	5 rpm
Cathode material	Aluminum

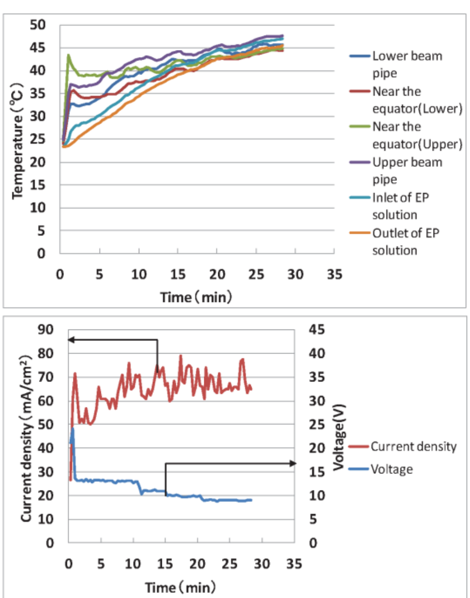

Figure 5: Upper: the plot of temperature vs. time. Lower: the plot of voltage and electric-current density vs. time in the first VEP experiment.

The conditions of experiment are described in Table 1. The measured temperature, voltage and electric-current density during the experiment are shown in Fig. 5 for the first VEP experiment. The temperature raised from 24 to 47 ^{0}C at the upper beam-pipe of cavity and this was the maximum. The electric-current density ranged from 50 to 80 mA/cm^2. The pictures of inner-surface of cavity before and after first VEP experiment are shown in Fig. 6. It is seen that the lower cup of cavity looked smooth but the upper cup looked rather rough. This might be the effect of bubbles during the VEP process.

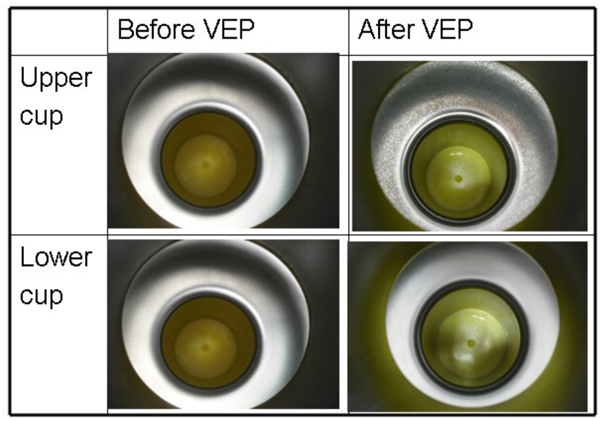

Figure 6: Pictures of inner-surface of cavity before and after the first VEP experiment.

Second VEP Experiment

We performed the second VEP experiment with the same Nb single-cell cavity. However, we set the upper cup of the cavity in the first VEP experiment to be the lower cup in the second VEP experiment. In other words, we flipped the upper side of cavity to lower from the first to the second VEP experiments.

Table 2: Conditions of second VEP experiment

Items	Condition
Electrolyte composition	H_2SO_4(98%) : HF(55%) = 9:1 (V/V) and fresh electrolyte
Voltage	9 V
EP duration	(EP for about 10 min. and switched off voltage for cooling) repeated 6 times
Flow direction	Upward (bottom to top)
Flow rate	5 L/min.
Cathode rotation speed	5 rpm
Cathode material	Aluminum

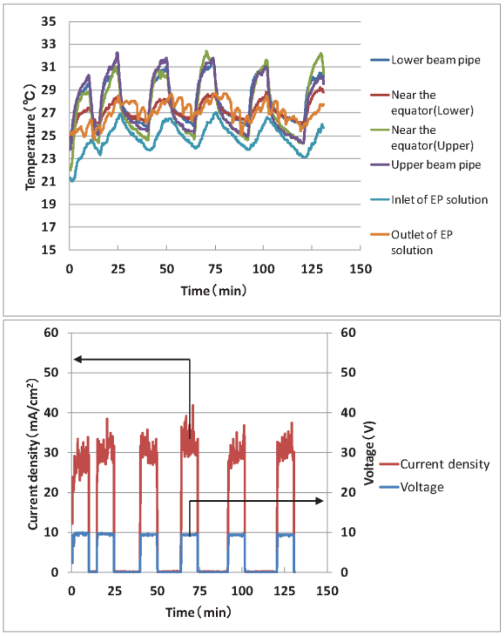

Figure 7: Upper: the plot of temperature vs. time. Lower: the plot of voltage and electric-current density vs. time in the second VEP experiment.

The sequence of the second VEP experiment was the same as of the first VEP experiment. The conditions of second VEP experiment are described in Table 2. The measured temperature, voltage and electric-current density during the second VEP experiment are shown in Fig. 7. In order to suppress the rise of temperature and the creation of bubbles in the VEP process, we reduced the voltage to 9 V and extended the total experimental duration from 30 min. to 60 min. with introducing the pause period to turn off the voltage about every 10 min.to wait for the decrease of temperature and the amount of bubbles. The temperature was successfully reduced and ranged from 23 to 32 ^{0}C, and the electric-current density ranged from 20 to 40 mA/cm^2 through the experiment.

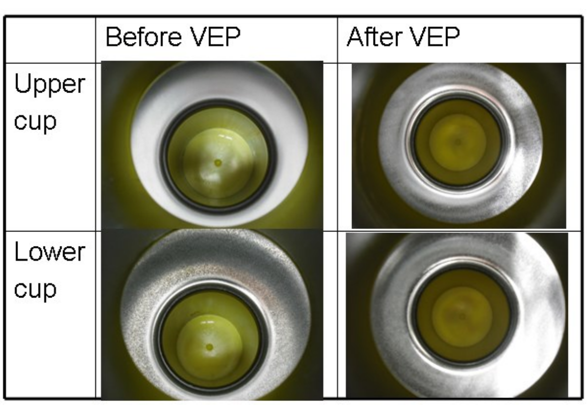

Figure 8: Pictures of inner-surface of cavity before and after the second VEP experiment.

The pictures of inner-surface of cavity before and after the second VEP experiment are shown in Fig. 8. It is seen that the upper cup of cavity looked smoother than after the first experiment, and the lower cup also looked smoother than after the first experiment. Thus on the both upper and lower cups are improved in terms of surface brightness and there were no traces of bubbles. This might be the effect of low temperature and small amount of bubbles during the second VEP process.

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Third VEP Experiment

We performed the third VEP experiment with the same Nb single-cell cavity. This time, we again set the upper cup of the cavity in the second VEP experiment to be the lower cup in the third VEP experiment. In other words, the posture of cavity was back to one in the first experiment. In the third experiment, in order to increase the effect of low temperature and small amount of bubbles, we added more functions to the setup to cool down the cavity and to suppress bubbles as shown in Fig. 9. The new setup includes (1) a spot cooler which was connected with a pipe to a bag surrounding the cavity and (2) the pump 2 which exhausts the bubbles in the upper beam-pipe by making a negative pressure on the upper surface of EP electrolyte and returned bubbles back to the EP electrolyte container.

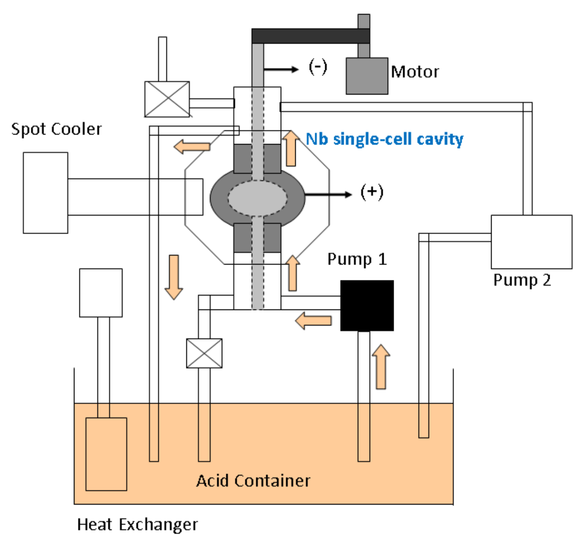

Figure 9: Schematic view of setup for the third VEP experiment.

The sequence of the third VEP experiment was the same as of the first and second VEP experiments. The conditions of the third VEP experiment are described in Table 3 where all parameters are kept the same as the second VEP process except for the aged EP electrolyte. The measured temperature, voltage and electric-current density during the third VEP experiment are shown in Fig. 10.

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Table 3: Conditions of third VEP experiment

Items	Condition
Electrolyte composition	H_2SO_4(98%) : HF(55%) = 9:1 (V/V) and aged in 2nd VEP
Voltage	9 V
EP duration	(EP for about 10 min. and switched off voltage for cooling) repeated 6 times
Flow direction	Upward (bottom to top)
Flow rate	5 L/min.
Cathode rotation speed	5 rpm
Cathode material	Aluminum

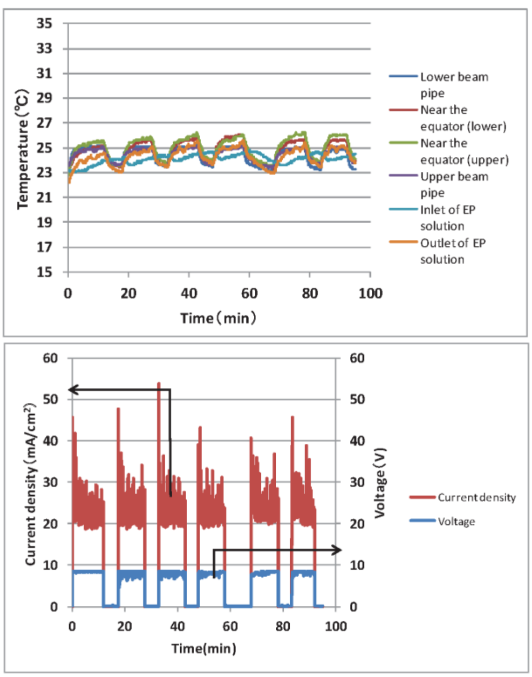

Figure 10: Upper: the plot of temperature vs. time. Lower: the plot of voltage and electric-current density vs. time in the third VEP experiment.

The temperature was successfully reduced again and ranged from 23 to 26 ^{0}C, and the electric-current density ranged from 20 to 35 mA/cm^2 except for very sharp peaks of oscillations. Also the amount of bubbles at the top of upper beam-pipe was clearly reduced being checked by eyes. The pictures of inner-surface of cavity before and after the third VEP experiment are shown in Fig. 11. It is seen that both the upper and lower cups of cavity looked similarly smooth but slightly looked like orange-peel surface, in particular near the equator.

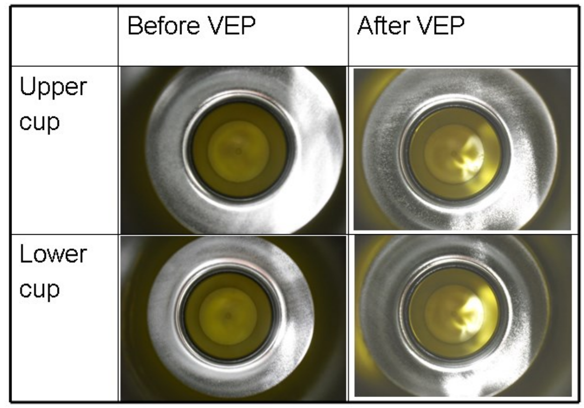

Figure 11: Pictures of inner-surface of cavity before and after the third VEP experiment.

Before and after the third VEP experiment, we measured the roughness on the inner-surface of upper and lower beam-pipes with a profilometer (Mitsutoyo SJ-301). The measured values of roughness are shown in Table 4. On the inner-surface of both beam-pipes, the measured roughness: Ra (arithmetic average), Ry (maximum height), and Rz (average over 10 points) are improved after the third VEP process. This confirmed the fact that the inner-surface looked smoother after the third VEP process.

Table 4: Roughness measurements before and after the third VEP experiment

		Before VEP	After VEP
Upper beam pipe (μm)	Ra	0.49	0.35
	Ry	2.8	2.1
	Rz	2.1	1.5
Lower beam pipe (μm)	Ra	0.48	0.37
	Ry	2.8	2.1
	Rz	1.9	1.4

We also measured the thickness of Nb material of cavity over 24 points before and after the third VEP process with ultrasonic thickness gauge (GE sensing & inspection technology, CL-5) calibrated by measuring a coupon of Nb plate of a known thickness. The measured points on the single-cell cavity are numbered as shown in Fig. 12. For each point, we measured the thickness three times and took the average before and after the third VEP process. Then the removal thickness by the third VEP process was estimated for each point and the results are shown in Fig. 13. The average of removal thickness over 24 points was 17 micrometer.

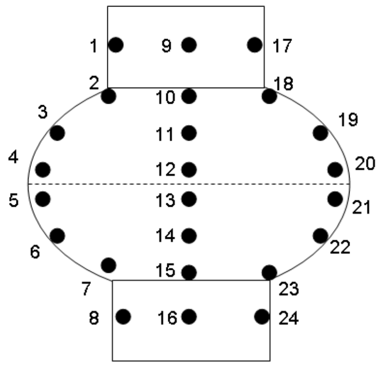

Figure 12: Locations of measured point on the single-cell cavity for the thickness measurements before and after the third VEP experiment.

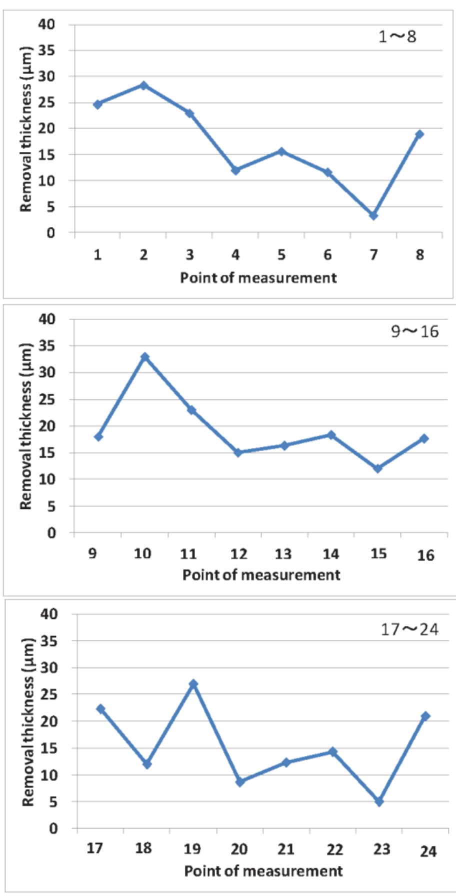

Figure 13: Removal thickness by the third VEP experiment at 24 points on the single-cell cavity. Upper: point 1 – 8. Middle: point 9 – 16. Lower: point 17 – 24. For each point, we measured the thickness three times and took the average before and after the third VEP, and the difference is plotted as the removal thickness.

If we see Fig. 13, the geometrical dependence of the removal thickness is not clear because rather large scattering is seen in measured values over 24 points. The scattering for the three measurements at each point was about 1 to 2 micrometer. If we consider the propagation of error in the subtraction formula, the scattering of removal thickness might be ranging from 1.4 to 3 micrometer. But more systematic error might be caused by contacting condition between the sensor head of gauge and the surface of the cavity. However even the error of measurements might be rather large, in simple comparison, the removal thickness seems thick in the beam-pipes (measured points: 1, 8, 9, 16, 17, 24) and thin near the equator of cell (measured points: 4, 5, 12, 13, 20, 21). This shows that enough uniformity for the removal thickness might not be achieved yet, and then the parameter-set of VEP process with i-cathode “Ninja” ® might need more optimization.

In the near future, we will repeat the VEP experiments of single-cell cavities continuously and will try to achieve the optimized parameter-set to realize smooth and uniform inner-surface and uniform removal thickness without geometrical dependence. We also have a plan to measure the gradient of single-cell cavity which is prepared with VEP process with i-cathode “Ninja” ®. Moreover, we are planning VEP experiments with Nb 9-cell cavities.

SUMMARY

We have performed the Vertical Electro-Polishing (VEP) experiments with a plastic mock-up of 9-cell cavity and a Nb single-cell cavity using the i-cathode “Ninja” ® which has retractable wings on the cathode rod to improve the uniform flow of electrolyte in the cavity and the uniform electrical field between the cathode and the cavity (anode). In the experiment with a plastic mock-up, we confirmed the effect of wings on the cathode to improve the uniform flow of electrolyte. In the three VEP experiments with a Nb single-cell cavity, the parameter-set of VEP process was improved step by step and the inner-surface of cavity and the removal thickness were reported. We are planning additional VEP experiments with Nb single-cell cavities and also Nb 9-cell cavities including the gradient tests of cavities in vertical cryostat (vertical tests).

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