R&D FOR THE POST-EP PROCESSES OF SUPERCONDUCTING RF CAVITY

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Abstract

The Electro-Polishing (EP) process is the best candidate of final surface removal for the production of ILC cavities. Nevertheless, the broad distribution of the gradient caused by field emission is still a serious problem for the EP process. One candidate of sources for the field emission is the sulfur component which is produced during the EP process. We studied the effect of post-EP rinsing treatments with ethanol, detergent, and H_2O_2 in various conditions by a unique method. Moreover, we tried to test sponge cleaning as a new post-EP process. This article describes the results of series tests of these post-EP processes.

INTRODUCTION

In the activities of Global Design Effort (GDE) to study the construction of International Linear Collider (ILC), the acceptant gradient of produced Superconducting RF (SRF) 9-cell cavities was determined to be 35 MV/m in vertical test [1]. The yield rate of the cavity beyond this threshold should be more than 90% to minimize the cost of ILC. However, even if choosing one qualified vendor, the current yield rate of produced cavities is only 45%.

In the standard recipe of ILC cavity treatment, the Electro-Polishing (EP) is the final process to remove the inner surface of the cavity. But the following post-EP rinse-processes; ultrasonic Ultra-Pure Water (UPW) rinse, ethanol-rinse, detergent-rinse and High Pressure Rinse (HPR) seem not to be enough to remove residual particles on the inner surface of cavity. This causes the field emission of cavities and also the resultant broad distribution of gradient. One candidate of field emitters produced in the EP process is the sulfur component. We found sulfur powder on the cathode-bag which is covering the cathode-rod in the EP facility at KEK. We collected the sulfur powder by a unique method and utilized it to test the effect of various post-EP processes. In addition, we are studying sponge-wipe cleaning as a new post-EP process. The plan of R&D for the sponge-wipe process is also described in this article.

EP FACILITY AT STF/KEK

We have constructed an EP facility in the Superconducting RF Test Facility (STF) at KEK in 2008. The EP facility has been used for the EP process of SRF 9-cell cavities for more than one year. The picture of the EP facility at STF/KEK is shown in figure 1. During the EP process, EP acid is pumped up from the reservoir tank of EP acid and flows through inside the 9-cell cavity and back to the reservoir tank again. In order to apply the voltage to the cavity, an aluminum cathode-rod is inserted through the cavity. When the voltage is applied between the cathode-rod and anode (cavity), hydrogen gas is produced on the surface of cathode-rod. The gas is collected by Teflon-mesh cathode-bag, which is covering the cathode-rod and is open to the both beam-pipe ends of cavity for the hydrogen gas to be exhausted. The cross-section of cavity, cathode-rod and cathode-bag are illustrated on the left-hand side of figure 2.

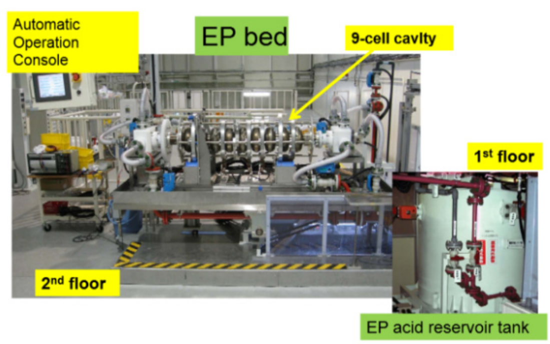

Figure 1: Pictures of the EP facility at STF/KEK. There is an EP bed on the 2nd floor and the reservoir tank of EP acid is on the 1st floor.

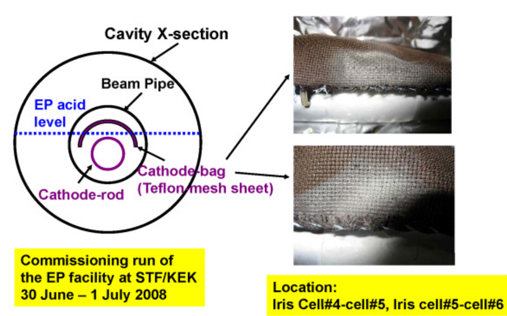

Figure 2: Left-hand side: The cross-section of cavity to illustrate the cathode-rod and the cathode-bag. Right-hand side: Pictures of the cathode-bag and the yellowish-white powder on it.

After some EP processes of 9-cell cavities during the commissioning of the EP facility from 30th June to 1st July in 2008, we found yellowish-white powder on the

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cathode-bag. The pictures of the cathode-bag and the powder on it are shown on the right-hand side of figure 2. The powder is mainly found at the position corresponding to the iris between cell#4 and cell#5 of 9-cell cavity.

ANALYSIS OF POWDER BY XRF AND X-RAY DIFFRACTION

The yellowish-white powder on the cathode-bag was collected by rinsing the cathode-bag with ethanol. The ethanol and powder were analyzed by X-ray fluorescence (XRF) analyzer. The result is shown in figure 3. The main component of powder was found to be sulfur (86%).

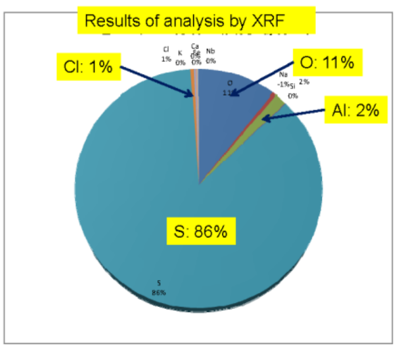

Figure 3: The analysis of ethanol and powder by X-Ray Fluorescence (XRF) analyzer. The main component of powder was found to be sulfur (86%). The rest of components were O (11%), Al (2%) and Cl (1%).

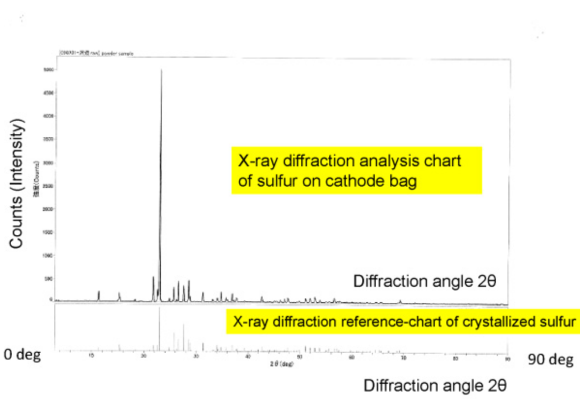

Figure 4: The analysis of ethanol and powder by X-Ray diffraction analyzer. Upper chart is for the powder and the lower chart is for the crystallized standard sulfur-sample.

In addition to the XRF analysis, we analyzed the powder by X-ray diffraction analyzer. The result is shown in figure 4, where the X-ray diffraction chart of the powder can be compared with that of the crystallized standard sulfur-sample. As seen in figure 4, the specific peaks in the chart of the powder coincide very well with the peaks of the standard sulfur-sample in the form of crystal. This shows that the sulfur component of the powder is also in the form of crystal.

ANALYSIS OF POWDER BY SEM AND EDS

In order to analyze the property of sulfur powder, we tried to collect the sulfur powder by setting detachable Teflon-mesh sheets on the cathode-bag as shown in figure 5. After some EP processes of 9-cell cavities had been performed at STF/KEK with these detachable Teflon-mesh sheets, we detached the Teflon-mesh sheets and observed them by a microscope. We found that a lot of particles are collected on the surface of Teflon-mesh fibers as shown on the right-had side of figure 5.

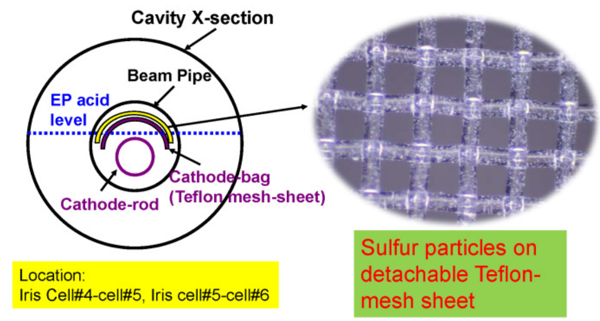

Figure 5: Left-hand side: The cross-section of cavity during EP process. Over the cathode-bag, detachable Teflon-mesh sheets were set in order to collect sulfur powder. Right-hand side: The picture of a detachable Teflon-mesh sheet by a microscope. Many particles of powder are seen as white dots on the surface of Teflon-mesh fibers.

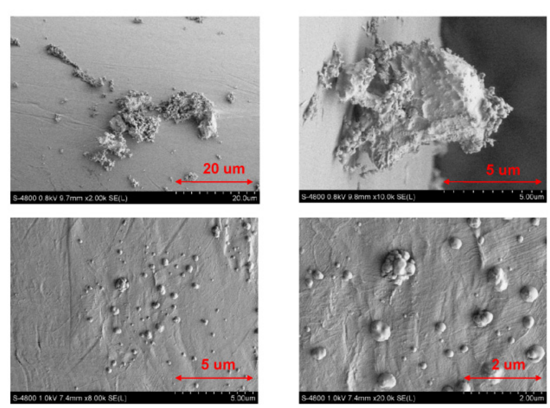

Figure 6: Images of particles on the detachable Teflon-mesh sheet by SEM.

The detachable Teflon-mesh sheet was observed by a Secondary Electron Microscope (SEM) too. Some images by SEM are shown in figure 6. As seen in the figure, the size of particles in the powder is ranging from sub-micron to a few 10's microns.

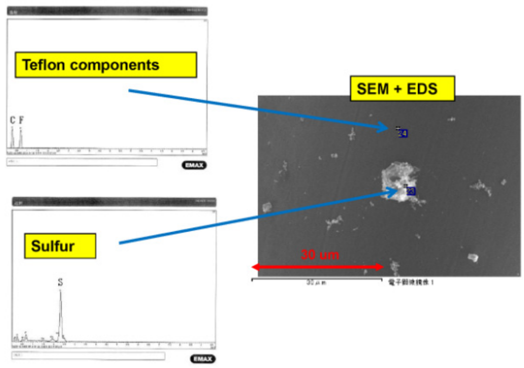

Figure 7: The results of EDS analysis for a particle on the surface of Teflon-mesh fiber. Lower left is EDS analysis on a particle and upper left is EDS analysis off the particle. The main component observed on the particle was sulfur. On the other hand, the main components observed off the particle were C and F which are the components of Teflon fiber.

In order to confirm if the particles observed by SEM has sulfur component, we analyzed the components of a particle by Energy Dispersive X-ray Spectrometry (EDS). The results are shown in figure 7. The main component observed on a particle was sulfur. On the other hand, the main components observed off particles were C and F which are the components of Teflon fiber.

We also tried to observe sulfur particles on the inner surface of cavity after EP process. Details of this study are found elsewhere [2].

RINSE TESTS WITH POWDER ON TEFLON-MESH SHEETS

In order to test the effect of post-EP rinse processes in various conditions, we utilized the detachable Teflon-mesh sheets with sulfur powder. We emphasize here that the sulfur powder on the detachable Teflon-mesh sheets were produced in the EP processes of real 9-cell cavities at STF/KEK. If the powder is removed from the sheets in a particular rinsing condition, the condition might be expected to be effective for the rinse of 9-cell cavity too, though there is a difference between Teflon-mesh sheet and niobium. The rinse conditions we tested are listed in figure 8. Two detergents, i.e. LION SUNWASH FM-20 and FM-550, ethanol, and H_2O_2 were used as solution. The parameters of condition were concentration, temperature, rinsing duration, and mechanical vibration methods, i.e. vibration at a few Hz or ultrasonic vibration.

Some examples of the test results are shown in figure 9. We first rinsed the Teflon-mesh sheet in Ultra-Pure Water (UPW) with ultrasonic vibration, but this rinsing method had almost no effect to remove the powder from the sheet. On the other hand, we found that ethanol-rinse with ultrasonic vibration and detergent-rinse (FM-20 with the concentration of > 2%, FM-550 with the concentration of >10%) with ultrasonic vibration perfectly remove the powder from the sheets, where the detailed parameters are described in figure 8.

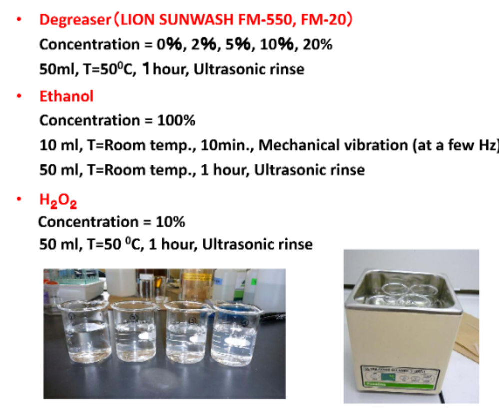

Figure 8: Upper: Various rinsing conditions of the tests. Lower left: Teflon-mesh sheets dipped in various solutions. Lower right: Rinse with ultrasonic vibration.

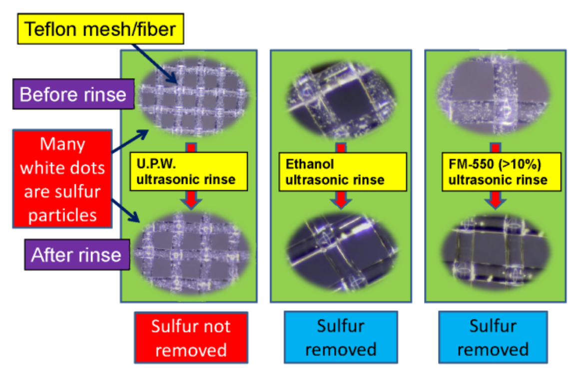

Figure 9: Some examples of test results. UPW rinse with ultrasonic vibration had almost no effect to remove sulfur particles on the Teflon-mesh sheet. Ethanol-rinse with ultrasonic vibration and FM-550 (>10%) with ultrasonic vibration removed all of sulfur particles on the Teflon-mesh sheet.

• Test of FM-20 (LION detergent) and 10% H_2O_2

	UPW Ultrasonic rinse	FM-20 2 % Ultrasonic rinse	FM-20 5 % Ultrasonic rinse	FM-20 10 % Ultrasonic rinse	H_2O_2 10% Ultrasonic rinse
Cleaning Result	×	○	○	○	× or △

• Test of Ethanol and FM-550 (LION detergent)

	Ethanol mech. vib. rinse	Ethanol ultrasonic rinse	FM-550 2 % Ultrasonic rinse	FM-550 5 % Ultrasonic rinse	FM-550 10 % Ultrasonic rinse	FM-550 20 % Ultrasonic rinse
Cleaning Result	△	○	△	△	○	○

Figure 10: Summary of the rinse tests. A circle mark means all sulfur particles were removed in the condition. A cross mark means most of sulfur particles remained after rinse in the condition. A triangle mark means some particles were removed in the condition. See figure 8 for the detailed parameters of conditions.

The results of rinse tests in various conditions are summarized in figure 10. An interesting result is that ethanol-rinse with mechanical vibration at a few Hz has much less effect than with ultrasonic vibration. H_2O_2 rinse with ultrasonic vibration was found to be not effective to remove the sulfur powder from the Teflon-mesh sheet.

R&D FOR SPONGE-WIPE PROCESS

We are studying sponge-wipe process as a new post-EP process to remove field emitters on the inner surface of cavity. We already fabricated a proto-type sponge-wipe tool for TESLA-shape single-cell cavity. The drawings and pictures of the proto-type sponge-wipe tool are shown in figure 11. Pieces of sponge are attached on the four blades which have extendable structure. The structure is inserted into a single-cell cavity and four blades are extended like umbrella, and the structure can be rotated by a handle to wipe the inner surface of cavity. Detailed description of the proto-type sponge-wipe tool and the first fitting test with a single-cell cavity can be found elsewhere [3].

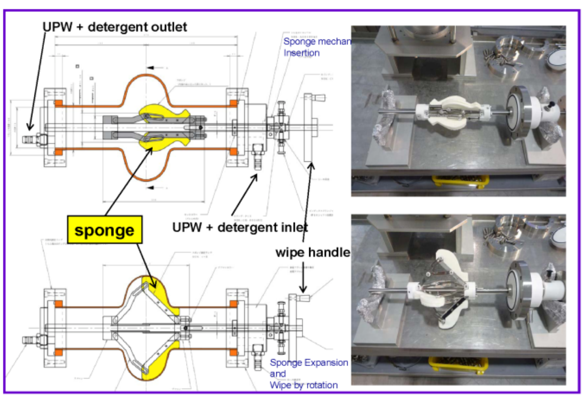

Figure 11: Left-had side: the drawings of the proto-type sponge-wipe tool. Right-had side: The pictures of the proto-type sponge-wipe tool.

In the first fitting test of the proto-type sponge-wipe tool, we found that a lot of particles are emitted from sponge material and these particles remain inside the cavity after the sponge-wipe process. We thought that bonding force between sponge-particles and cavity surface might be very weak and these sponge-particles might be easily removed by HPR. In order to confirm if the sponge-particles can be removed by HPR and also the sponge-wipe process itself is harmless to a SRF cavity, we made a plan of experiments in collaboration between KEK and CEA/Saclay. In this plan, we selected a TESLA-shape single-cell cavity (D1DE1) which reached the gradient of 40 MV/m in last vertical test at CEA/Saclay in October 2008. If the sponge-wipe and HPR are performed on the cavity and the gradient of the cavity is kept at 40 MV/m in following vertical test, we could conclude that the sponge-wipe process itself might be harmless to the performance of SRF cavity. The cavity (D1DE1) was shipped from CEA/Saclay to KEK in July 2009. The sponge-wipe experiment of cavity was done in August 2009. However, during the sponge-wipe process, the central rod of the sponge-wipe tool was disassembled inside the cavity due to bad design of sponge-wipe structure. In the result, the iris part of the cavity (D1DE1) was scratched by one of the four blades of sponge-wipe tool. The cavity (D1DE1) was shipped back to CEA/Saclay to cure the scratch at the iris by Buffered Chemical Polishing (BCP). Also the design of sponge-wipe tool was re-examined and modification was applied to the proto-type sponge-wipe tool. We have the plan to cure the cavity and confirm the performance of cavity (D1DE1) by vertical test at CEA/Saclay. We will repeat the experiment with the cavity again.

In addition, we have the plan to test the proto-type sponge-wipe tool with the cavity which is suffered from field emission. We already selected the cavity candidate (C1-21) in collaboration between KEK and CEA/Saclay. The last vertical test of the single-cell cavity (C1-21) is shown in figure 12. In this plan, we will perform a sponge-wipe on the cavity (C1-21) at KEK and perform a vertical test of the cavity at CEA/Saclay to see the effect of sponge-wipe process.

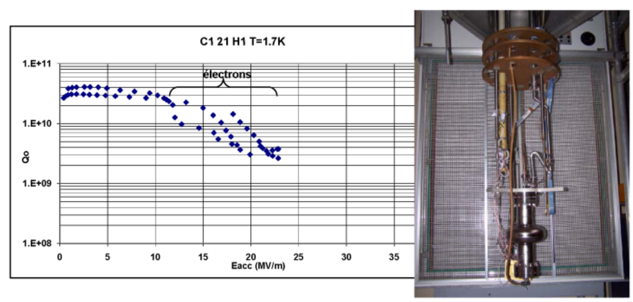

Figure 12: Left-hand side: The result of last vertical test for the single-cell cavity (C1-21) at CEA/Saclay. The Q0 vs. Eacc plot shows that the performance of the cavity is limited by field emission above the gradient of 10 MV/m. Right-hand side: The picture of vertical test stand for a single-cell cavity at CEA/Saclay.

CONCLUSIONS

In the commissioning of new EP facility at STF/KKE in 2008, we found white-yellowish powder on the cathode-bag which is covering the EP cathode-rod. The XRF analysis of the powder showed that the main component of powder is sulfur (86%) and the analysis by X-ray diffraction analyzer showed that the sulfur powder is in crystal form. We collected the powder with detachable Teflon-mesh sheets on the cathode-bag during EP process at STF/KEK, and observed the detachable Teflon-mesh sheets by SEM and EDS. As the results, we found that the size of particles in the powder is ranging from sub-micron to a few 10's microns, and the main component of the particle in powder was confirmed to be sulfur. The detachable Teflon-mesh sheets with powder were utilized to test various post-EP rinsing methods. The results of tests showed that ethanol-rinse with ultrasonic vibration, detergent rinse (FM-20 with > 2% and FM-550 with > 10%) with ultrasonic vibration are effective to remove the powder from the sheets. On the other hand, ethanol-rinse with mechanical vibration at a few Hz, detergent-rinse (FM-550 with < 10 %) with ultrasonic vibration, and H_2O_2 rinse (10%) with ultrasonic vibration were not enough to remove the powder from the sheets.

We are studying sponge-wipe process as a new post-EP process and already fabricated a proto-type sponge-wipe tool for TESLA-shape single-cell cavity. We newly started the activity of sponge-wipe study in collaboration between KEK and CEA/Saclay. In the activity we performed sponge-wipe process on a single-cell cavity having a good performance in last vertical test. This test was intended to confirm if the sponge-wipe process itself is harmless to the performance of SRF cavity. However, the wipe test failed due to bad design of sponge-wipe tool. The design was modified according to the re-examination of the design and the modification was applied to the sponge-wipe tool. We have the plan to perform sponge-wipe test with a single-cell cavity with a good performance again, and moreover, a single-cell cavity suffered from field emission. In the latter test, we might be able to study the effect of the proto-type sponge-wipe tool against field emission in the near future.

ACKNOWLEDGEMENT

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REFERENCES

[1] ILC RDR, http://lcdev.kek.jp/RDR/.

[2] X. Zhao, et al., “Surface characterization of niobium samples electropolished together with real cavities”, this proceedings, TUPPO086; M. Nishiwaki, et al., “Surface study using niobium sample coupons for superconducting RF cavity”, PAC’09, Vancouver Canada, May 2009, TU5PFP070.

[3] T. Saeki, et al., “R&D for sponge cleaning of super conducting RF cavity”, PAC’09, Vancouver Canada, May 2009, TU5PFP072.