

STUDY ON ELECTRO-POLISHING PROCESS BY NIOBIUM-PLATE SAMPLE WITH ARTIFICIAL PITS

T. Saeki*, H. Hayano, S. Kato, M. Nishiwaki, M. Sawabe, KEK, Tsukuba, Japan
 W. Clemens, R. L. Geng, R. Manus, JLAB, Newport News, U.S.A.
 P. V. Tyagi, GUAS/AS/KEK, Tsukuba, Japan

Abstract

The Electro-polishing (EP) process is the best candidate of final surface-treatment for the production of ILC cavities. Nevertheless, the development of defects on the inner-surface of the Superconducting RF cavity during EP process has not been studied by experimental method. We made artificial pits on the surface of a Nb-plate sample and observed the development of the pit-shapes after each step of 30um-EP process where 120um was removed by EP in total. This article describes the results of this EP-test of Nb-sample with artificial pits.

INTRODUCTION

The bad performance of Superconducting RF (SRF) accelerating cavities are mainly caused by quench and field-emission. One of candidates to cause the quench is the existence of visible defects on the inner-surface of cavity. Recently, the Electro-polishing (EP) process is the best candidate of final surface-treatment for the high accelerating-gradient cavity. However, there is a natural question that if the EP process is enough to cure the visible defects on the inner-surface of cavity. In such situation, there has been no systematic study on the development of defects on the inner-surface of the SRF cavity during EP process by a clear experimental method. In order to answer this question, we performed EP process on a niobium (Nb) plate sample which has some artificial defects/pits on the surface, in the collaboration of KEK and JLab. We performed the EP process step by step with removal thickness of 30 um by 4times, i.e. 120 um in total. In each step, the development of pit-shape was observed by a laser optical micro-scope. This article reports the results of this experiment.

NIOBIUM-PLATE SAMPLE WITH ARTIFICIAL PITS

The preparation of Nb-plate sample with artificial pits was done in JLab. Many hard alumina micro-balls in the size ranging from a few tens to a few hundreds microns were pressed on the surface of Nb-plate sample. The alumina balls were embedded into the surface of the Nb-plate sample and these balls were molten in following Buffered Chemical Polishing (BCP) process. After melting all alumina balls, pits were created on the surface of the Nb-plate sample. The Nb-sample was sent from JLab to KEK. A picture for the Nb-plate sample with these artificial pits is shown in Fig. 1. We made four scratching lines around pits to make it easier to identify

the position of each pit. Then we named seven pits as pit 1 – 7 as shown in Fig. 1.

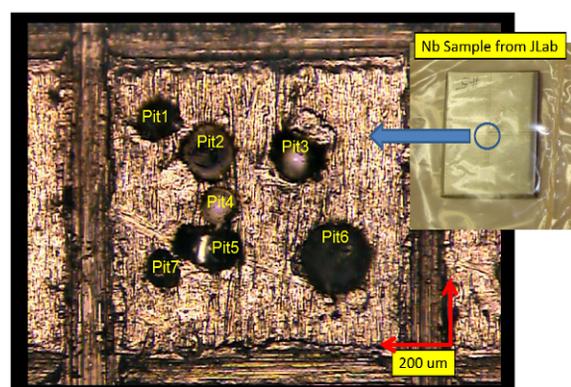


Figure 1: A picture on the surface of the Nb-plate sample with pits by an optical micro-scope.

LASER OPTICAL-MICROSCOPE

We utilized a laser optical-microscope: KEYENCE VK-8500. We could take optical micro-scope images in the format of digital files by this tool. And we could also make 3-Dimensional (3D) contour plot of observing surface of object by laser scanning with changing the focus of optical system automatically. Pictures of the laser optical micro-scope are shown in Fig. 2.

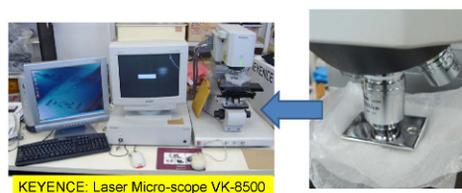


Figure 2: Pictures of the laser optical micro-scope: KEYENCE VK-8500.

BCP PROCESS OF NIOBIUM-PLATE SAMPLE

After receiving the Nb-plate sample at KEK, all pits were observed by the laser optical micro-scope. As the result, we found some small alumina balls were still remaining at the bottom of some pits. We added BCP process by 30 um to remove the alumina balls completely and also this cleaned up the surface of Nb-plate sample before EP process. The BCP process was done at KEK

*takayuki.saeki@kek.jp

and then the 3D shape of each pit was analyzed by the laser optical micro-scope as shown in Fig. 3.

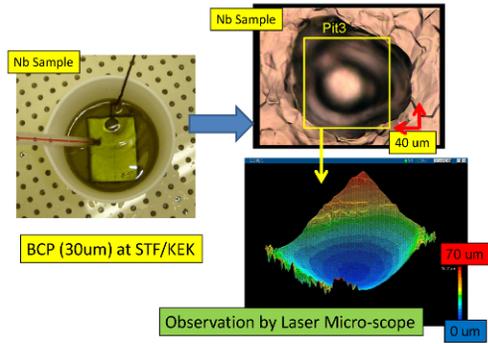


Figure 3: Left-hand side: Nb plate-sample was Buffered Chemical Polished (BCP'ed) by 30 um. Upper right-hand side: The optical image of the pit 3 after BCP(30 um). Lower right-hand side: 3-Dimensional (3D) contour plot for the pit 3 by laser optical micro-scope.

EP PROCESS OF SAMPLE

After BCP (30 um) at KEK, we performed EP process of the Nb-plate sample at Nomura plating Corp. The EP was performed progressively by the four steps where the removal thickness of each step is 30 um. Consequently, the total removal thickness of EP processes was 120 um. The setup of the EP process is shown in Fig. 4. A picture of EP setup is shown in Fig. 5. After each step of EP process, the sample was rinsed with Ultra-Pure water as shown in Fig. 5.

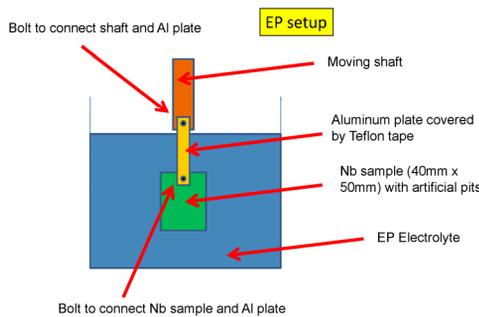


Figure 4: The schematic of setup for the Electro-Polish (EP) process of the Nb-plate sample.



Figure 5: Left-hand side: A picture for the EP process of Nb-plate sample. Right-hand side: A picture for the Ultra-Pure Water (UPR) rinse after each EP process.

OBSERVATION OF PIT-SHAPE BY LASER OPTICAL-MICROSCOPE

After each step of EP (30 um), the 3D shapes of all pits were analyzed by the laser optical micro-scope. The optical images of pit 3 after each BCP and EP process are shown in Fig. 6.

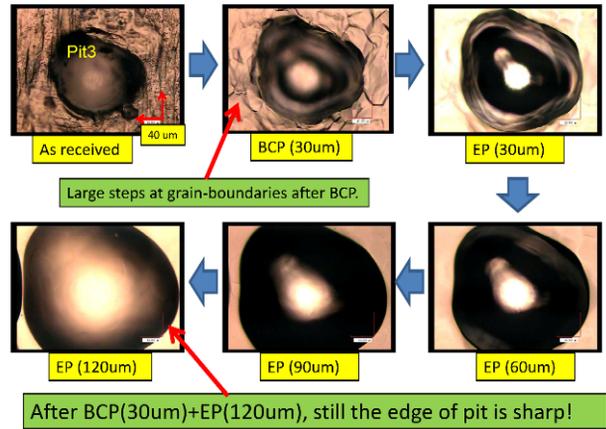


Figure 6: Optical images of pit 3 for as received, after BCP(30 um), EP (30 um), EP (60 um), EP (90 um), and EP (120 um).

As seen in the second image (BCP) in the figure, we found clear steps along the grain-boundaries on the surface of sample after BCP (30 um). On the other hand, the steps along the grain-boundaries became much smoother after progressive EP processes. However, the edges of all pits have not been rounded by EP process. Even after EP (120 um), still sharp edges of pits had been observed. This result indicates that the EP process is not effective and not perfect to remove the sharp edges of defects.

UNIFORM REMOVAL MODEL

In order to understand the removal feature of EP process clearly, we firstly considered a simple model of removal in which all the surface of sample is removed uniformly. We named this model as a uniform-removal model. A schematic for explaining this model is shown in Fig. 7. In the figure, the blue curve shows the original shape of a pit and the yellow curve shows the removed shape of the pit. If the removal is uniform and is independent of the geometrical shape of surface, the depth of the pit might be kept unchanged and constant. This is because the removal rates at the bottom of pit and on the flat surface are uniform. Moreover, we can calculate the development of the radius $R(t)$ as the function of removal thickness (t) by simple geometry/mathematics. The result is:

$$R(t) = \sqrt{R_0 \times R_0 - 2 \times R_0 \times t} \quad (1)$$

where $R_0 = R(t=0)$, i.e. the radius of original pit. Therefore, in this model, the radius of a pit increases as

the EP process proceeds, while the depth remains the same, and the edge of pit becomes dull very slowly.

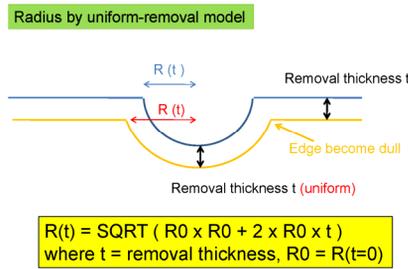


Figure 7: A schematic for the uniform-removal model. The blue curve shows the original shape of a pit. The yellow curve shows the resultant shape of the pit after uniform removal of thickness t um. The radius of pit is expressed as the function of thickness t and $R_0 = R(t=0)$.

DEVELOPMENT OF RADIUS AND DEPTH OF PITS IN EP PROCESS

After each EP process of 30 um, we measured and recorded the 3D shapes of all pits. The development of radius and depth of pit 3 is summarized in a plot shown in Fig. 8. In the plot, measured radius and depth of pit 3 are plotted as the function of removal thickness t (um), as well as the radius for the uniform-removal model. The measured radius and depth are the averages of several measurements for several different cross-sections of pit 3 with different rotating angle on the central/vertical axis of pit 3. This is because the shape of pit is not an ideal hemisphere shape.

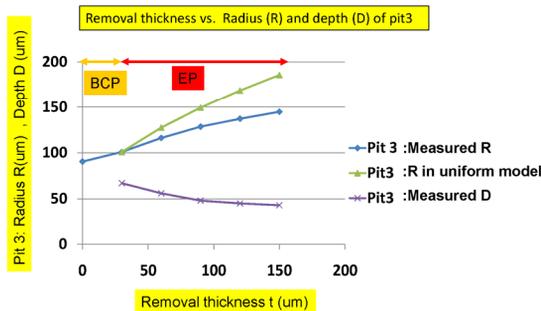


Figure 8: A plot for measured radius / measured depth of pit 3 vs. removal-thickness t (um) with blue and purple curves. Green curve shows the radius vs. removal thickness t in the case of the uniform-removal model.

The plot shows that the depth of pit decreases as the EP process proceeds (purple curve), and the developing speed of measured radius (blue curve) is less than that of the uniform-removal model (green curve). This indicates that the removal rate at the bottom and the edge of pit, i.e. inside pit, is less than that on the flat surface. Moreover, we plotted the measured radius vs. measured depth of pit 1 -7 as shown in Fig. 9. In this plot, Radius (R) and Depth (D) are measured only one time to simplify the analysis.

The plot shows again that the removal feature of real EP process is different from the uniform-removal model, because the measured ratio: dD/dR is roughly about 0.5 while the Depth (D) should be constant in the uniform-removal model.

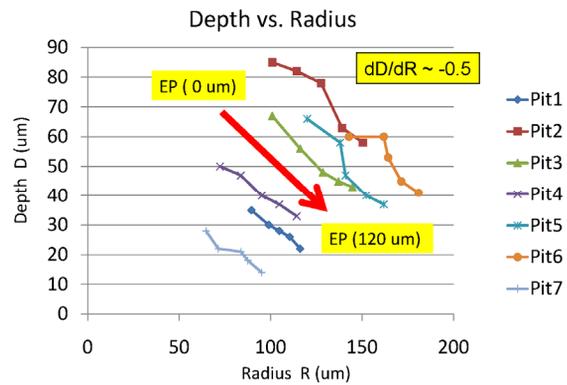


Figure 9: A plot for measured Depth (D) vs. measured Radius (R) of pit 1 – 7 for each EP removal of 0 um, 30 um, 60 um, 90 um, 120 um. The ratio: dD/dR is about 0.5.

DISCUSSIONS

It is found from this experiment that, when defects are caused by mechanical stress by pressing contaminant hard objects on inner surface of SRF cavities, the EP process is not enough and not almighty to cure the sharp edge of the defects. However, there is still possibility that the removal feature of EP might depends on how the defects are created. For example, it is interesting to perform the annealing of sample to release the mechanical stress of artificial pits before EP process. In recent studies of optical inspection of inner surface of SRF cavities, most pits are found in the Heat Affected Zone (HAZ) of Electron Beam Welding (EBW) seams. It is also interesting if a similar experiment is performed with an EBW'ed Nb-plate sample having pits in the HAZ of EBW seam.

SUMMARY

We made artificial pits on the surface of a Nb-plate sample and observed the development of the 3D shapes of pits by a laser optical micro-scope during progressive EP processes. The experimental result showed that the EP process is not enough and not effective to remove/cure the sharp edges of all pits. The removal rates in pits were found to be less than that on the flat surface of the sample. However, there are some possibilities that the experimental result depends on the way how pits are created on samples. Additional experiment is encouraged with an annealed sample to release the mechanical stress of artificial pits and/or a sample with pits in the HAZ area of EBW'ed samples.