STUDY ON THE BUFFERED ELECTROPOLISHING JACQUET LAYERS ON NIOBIUM CAVITY

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Abstract

Buffered electropolishing on niobium cavities has been developed at Peking University. Some small niobium samples have been polished very smoothly. An experiment has demonstrated that both of the liquid and solid Jacquet layers exist on the surface of anode simultaneously. The results of experiment, that more than 70% voltage is taken by the Jacquet layers, show us that the cathode shape is not a necessary condition for polishing the dumbbell. Flat cathode is available to polish the dumbbell. We have observed though the experiment that the different fluid liquid Jacquet layer movements could cause the different results on the dumbbell surface. These studies show that BEP can offer a smooth surface on niobium cavities.

INTRODUCE

The PKU-ERL-FEL will be based on superconducting 1.3 GHz nine-cell cavities of TESLA type. The surface condition of Nb superconducting radio frequency cavities is one of the most important factors in determining their accelerate grads. Traditional Buffered chemical polishing (BCP) is widely used in many laboratories because of easily. However BCP treatment will produces an enhanced etching rate at Nb grain boundaries and lead to rough Nb surface.

Electropolishing (EP) has been proven a necessary surface processing method for high-gradient superconducting niobium cavities [1]. EP using an acid mixture of H_2SO_4 and HF have been developed many labs. At the same time a new recipe including lactic, sulfuric and hydrofluoric acids have studied at Jlab, and has showed promising results [2, 3]. This process is called buffered eletropolishing.

We have got very small roughness Nb sample after optimizing the recipe and polishing condition. Furthermore, I design an experiment to study the character of Jacquet layers which play an important role on polishing the Nb. The device for polishing the dumbbell has been set up, the result of electropolishing on Nb dumbbells will report in this article.

EXPERIMENTS

The flat sample setup

The most important parameters of BEP are current density and voltage. Those two parameters are determined by I-V curve. We choose high pure Al as cathode material to do the BEP process [4]. The proportion of acid we used is hydrofluoric (48%): sulfuric (98%): lactic acids (85%) =5:4:11. The device of small Nb sample BEP treatment schematically is shown in Fig.1. The variation current can be read though change the voltage of electrical source. Thermometer can measure the temperature of the acid at any moment, at the same time; we can alter the outside temperature of the water with ice to influence the inside. With a magnet stir bar, the flowing velocity can be easily controlled.

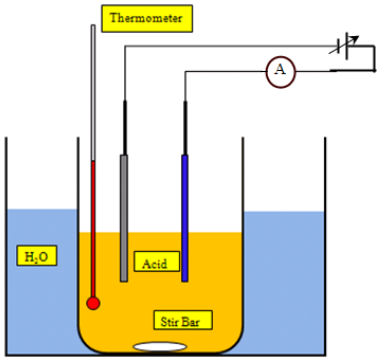

Fig.1. Schematic set-up of BEP for small samples

Study on Jacquet layers

P.A.Jacquet invent EP method at 1935, he found that there will be generate a liquid layer near anode [5]. Liquid layer which is a new viscous complex salt play a very important role in EP process. Lactic acids will electrochemical react with Nb and generate complex compound which is the obvious differences between EP and BEP. For study this complex compound how to affect the Nb surface, we design an experiment to measure some characters of complex compound. The experiment is schematically shown in Fig.2.

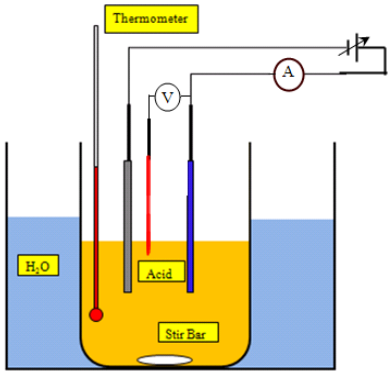

Fig.2. Schematic experiment of study Jacquet layers

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Resistance of complex compound is so larger than acid's. Therefore we make a probe immerging in the acid to measure the voltage between the probe and anode in the several different conditions.

Treatment for dumbbell

The device of EP treatment for 9-cell superconducting accelerator is very complex, and that will spent more time on arranging the device. So developing the BEP treatment for dumbbells has many advantages. The device is much easier to polish the dumbbell compare with 9-cell cavity, at the same time this step can save many times compare with EP treatment for 9-cell cavity. We use the high pure Al stick which diameter is 3cm as the cathode and let the Teflon cover the middle of stick about 1 cm. The device is present in the fig.3. The dumbbell set vertically. The acid can flows from the open tube with pump circularly. The Teflon net covers the Al stick to obstruct the air bubble which generator from cathode.

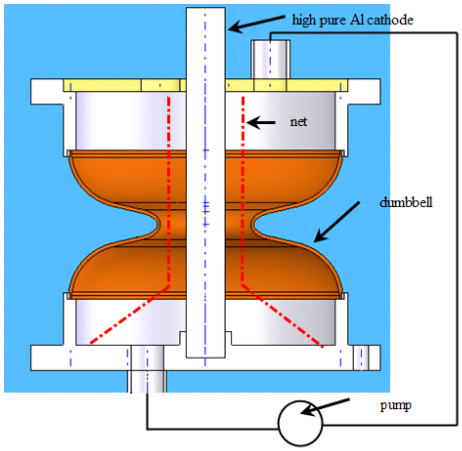

Fig3 Schematic device of BEP for dumbbell

RESULT AND DISCUSSION

Determination of fundamental polishing parameters

In the BEP procession, we can control some parameters like proportion of kind of acid, voltage, temperature, stirring speed. Current density is confirmed when we confirm these parameters. A typical measured I-V curve for the samples was shown in Fig.4. In the I-V curve, four regions with corresponding to etching, periodic oscillation of anode current density, polishing, and gas evolution on the anode surface were identified [4]. Four areas are identified as discussed in detail in reference [3].The work voltage point is determined by middle of polishing region. The temperature is below 30°C, since preventing such more hydrogen to filter into the Nb. High stirring speed will lead high current density as well as high speed of polishing, but the voltage confirming is difficultly. Through adjust the condition of polishing like stirring speed and voltage point, the speed of polishing can change from 0.55um/min to 3.79um/min when the current density change from 29mA/cm^2 to 235mA/cm^2, and all get very good sample surface. Fig5 shows typical optical images of surface, MOM photo in 1000 times of Nb sample surface and AFM photo with roughness data. We get the best RMS roughness 5.47nm at area of 400um^2. These results show that the surface polished by BEP is smoother than EP.

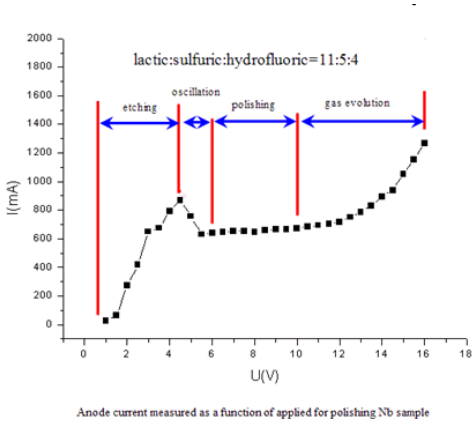

Fig. 4 Current density measured as a function of applied voltage for the polishing system

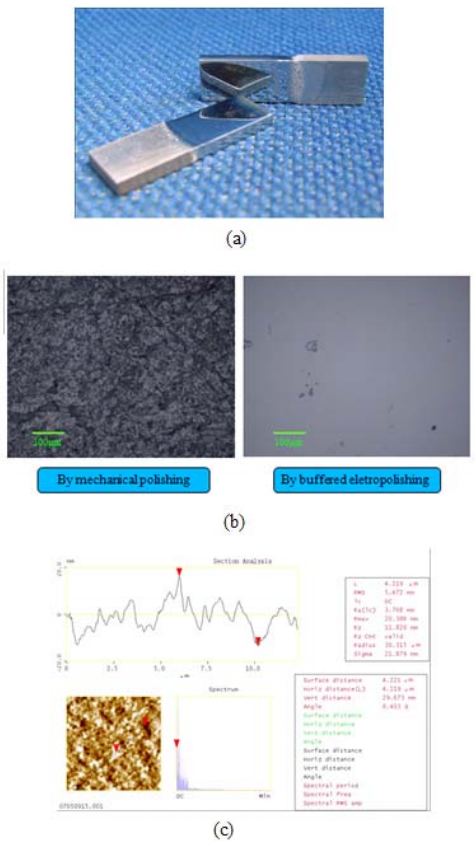

Fig.5.a) the optical images of surface .b) MOM photos in 1000 times of different Nb sample surface by mechanical polishing and by BEP. c) Nb sample surface profiles using AFM

Character of Jacquet layers

Through alter the distance of the probe and cathode; we can measure the voltage between them. The resistance of Jacquet layers is much larger than acid, so the resistance can represent such layers. In our experiment, the voltage of anode and cathode is 10V; the distance of pole keeps 30mm constantly and stirring speed much fast. The data present at the green line of Fig.6. And then moving anode keep the distance of pole 40mm. the black line show this condition. Due to the flowing acid have much effect to forming Jacquet layers, we slow down the stirring speed. This red line shows this condition which is much different with the black line. The blue line is the surface of anode. That the end point on the blue line is not 10V reveal that there is a solid layer on the surface of Nb. Long distance of two poles and slowing stirring speed all will lead more solid lay adhere on the Nb surface. On the one hand, the picture shows there are two different layers which called solid lay and liquid lay on the Nb surface. On the other hand, the liquid lay is transfer from the solid lay, because the sum voltage taking on the two Jaquet layers is 7V constantly in the different conditions.

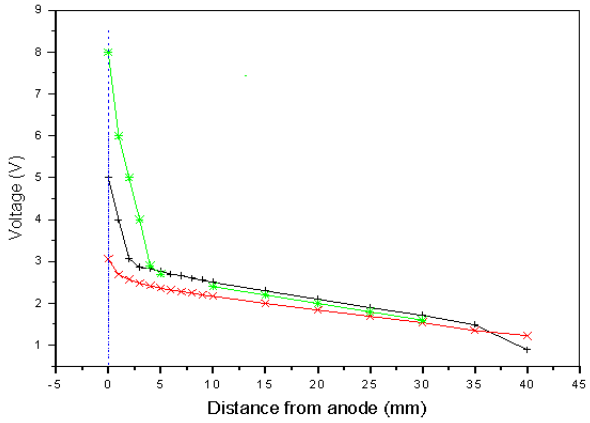

Fig.6. the voltage between probe and anode

According to the polishing theory, peak surface anode current concentrates more than bottom because of thin layers which take less voltage [4]. Comparing the voltage taking on the acid, we believe that the changes of distance of two poles are not affecting the electrical field on the surface. It demonstrates that the Al stick as cathode is fit for polishing the dumbbell. It is not necessary to use special shape of cathode which has a more uniform distance between cathode and anode. At the same time, this picture shows us the stirring speed should not so fast, because it will rush the liquid layers which is important for polishing. However, if the stirring speed is very slow, the acid near the anode surface will be diluted. So it will affect the speed of polishing and affect the surface roughness.

Polishing the dumbbell

We get very smooth surface on the up-bowl, however the line region of down-bowl show some grooves. The dumbbell polished show fig.7.There are not grooves on the iris region which is different from the result of article [2]. The Al stick can not support enough area to react to generator H_2, so we can not find a good work point on the I-V Curve. The influence of the cathode area to I-V curve is show in fig.8. Consequently, the flat Al combine with Al stick cathode is available to polish the dumbbell. The result of dumbbell reveal that the liquid Jacquet layer can be generated and keep on the up-bowl, however it can not stand under the down-bowl because of gravity. The vertical flow of acid will also easy wash out part of the liquid layers. The phenomenon of polished dumbbell is consistent with our judgment: no Jacquet layer, no polishing. At the same time, the movement of Jacquet layers will cause the different results on the dumbbell surface. The reason why to generator the groove is that the Jacquet layers under the down-bowl surface will be redistribution in the gravity field. Enough ice water retain outside of the dumbbell to keep the temperature not rise fast. However, controlling the acid temperature also is a problem in polishing the dumbbell. The most difficult is polishing the equator which has much impurity because of device structure.

Fig.7.the up-bowl of polished dumbbell

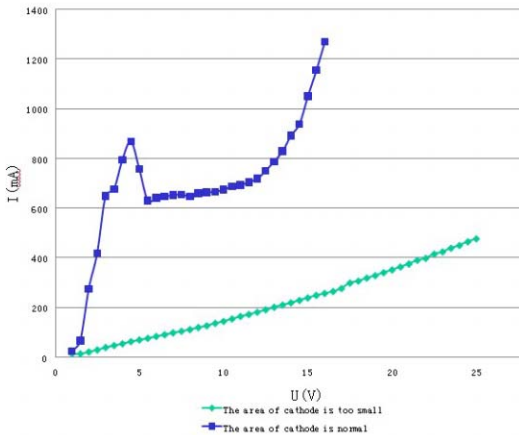

Fig 8. I-V curves with different cathode areas, the blue curve is normal. Much small area of cathode let the green curve like a line

CONCLUSION

Through study the BEP process, we collect enough data of Nb sample in different conditions. The mechanism of BEP had been research through measure the character of Jacquet layers. A rough model based on hydrodynamics and electrochemistry is introduced attempting to explain the generation process of some different results. Electropolishing the dumbbell is another technique to make the 9cell cavity. These study shows that BEP on the dumbbell is a promising technique for doing 9cell TESLA cavity.

ACKNOWLEDGMENTS

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REFERENCES

[1] K.Saito,in:Proceedings of the 2003 Partical Accelerator Conference,Portland,OR,USA2003,p,462.
[2] Jean R. Delayen et al. alternate electrolyte composition for electropolishing of niobium surfaces.
[3] Andy. T. Wu et al. Smooth Nb surfaces fabricated by buffered electropolishing. Applied Surface Science 253 (2007) 3041–3052
[4] J.R. Delayen, J. Mammosser, L. Phillips, A.T. Wu, in: Proceedings of the 10th Workshop on RF Superconductivity, Japan, (2001), p. 499.
[5] Jacquet, P.A. Trans. Electrochem Soc, 69, 629, (1936)