SURFACE TREATMENTS OF NB BY BUFFERED ELECTROPOLISHING*

A.T. Wu+, R.A. Rimmer, X.Y. Lu[1], F. Eozenou[2], C. Reece, L. Lin[1], G. Ciovati, J. Mammosser[3], S. Jin[1], E.D. Wang[1], J. Williams, and R. Manus
Thomas Jefferson National Accelerator Facility, 12000 Jefferson Avenue, Newport News, VA 23606, USA
[1] SRF Group, Institute of Heavy Ion Physics, School of Physics, Peking University, Beijing 100871 P. R. of China
[2] CEA, IRFU, SACM, LESAR Centre de Saclay, F-91191 Gif-sur-Yvette, France
[3] Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831, USA

Abstract

Buffered electropolishing (BEP) is an Nb surface treatment technique developed at Jefferson Lab [1]. Experimental results obtained from flat Nb samples show [2-4] that BEP can produce a surface finish much smoother than that produced by the conventional electropolishing (EP), while Nb removal rate can be as high as 4.67 μm/min. This new technique has been applied to the treatments of Nb SRF single cell cavity employing a vertical polishing system [5] constructed at JLab as well as a horizontal polishing system at CEA Saclay. Preliminary results show that the accelerating gradient can reach 32 MV/m for a large grain cavity and 27 MV/m for a regular grain cavity. In this presentation, the latest progresses from the international collaboration between Peking University (PKU), CEA Saclay, and JLab on BEP will be summarized.

INTRODUCTION

Surface smoothness is one of the critical parameters that affect the performance of Nb SRF cavities. Experiment has demonstrated that Nb SRF cavities with a smoother surface finish have more chances to reach high accelerating gradient [6]. We believe that following two reasons are mainly responsible for the experimental result. A) Smoother surface finish allows a relatively easy removal of contaminants from the inner surfaces of Nb SRF cavities, leading to a less chance of the Q degradation due to field emission. B) There will be less serious effect from the enhancement of RF magnetic field [7] on the surface. It has been shown in reference 8 that a surface roughness of less than 2 μm is required in order to achieve an accelerating gradient larger than 30 MV/m for Nb SRF cavities.

BEP is a verified Nb surface polishing technique that can produce the smoothest surface finish on Nb [1-4, 9-10]. Logically one wants to know how Nb SRF cavities will perform after BEP treatments. Recently, a simple and reliable vertical electropolishing (EP) system [5] was constructed at JLab to do treatments of Nb SRF single cell cavities by BEP. Several single cell cavities were treated by BEP both vertically [2] and horizontally of CEA Saclay [11]. Accelerating gradients of 32 MV/m was reached for a large grain cavity and of 27 MV/m for a regular grain cavity. In this report, we will summarize the latest progresses made on BEP from the international collaboration between PKU, CEA Saclay, and Jefferson Lab.

DEMOUNTABLE CAVITY

Our experience with the EP of Nb cavities so far has told us that polishing parameters obtained from small flat samples cannot be simply applied to the treatments of Nb cavities. Due to the complicated geometry associated with the anode of an Nb cavity and a pure Al cathode, it was very difficult to find an ideal I-V curve with a well defined polishing plateau. Therefore surface finish of an BEP treated Nb cavity is often not the same as those obtained from small flat samples. To study how the polishing parameters affect surface finish, a demountable cavity as shown in Fig.1 was designed and fabricated at JLab. Three Nb button samples can be mounted at locations close to equator, midway between the equator

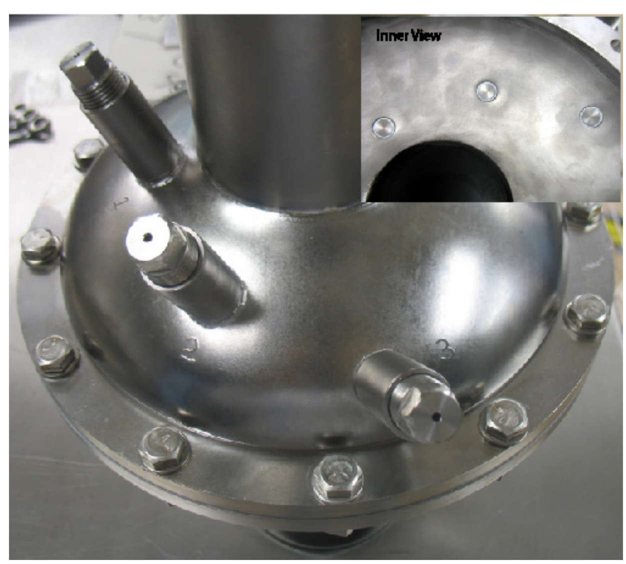

Figure 1: Demountable cavity for BEP study.

*Work supported by the U.S. Department of Energy, contract DE-AC05-84-ER40150.

+ andywu@jlab.org

and iris, and close to iris. The Nb buttons are processed together with the cavity and then they can be removed for various analyses. For consistency, the buttons were made from the same Nb batch. The buttons are also electrically insulated from the cell to allow the measurements of I-V curves at the three locations. After the fabrication, this demountable cavity was shipped to PKU for doing experiments.

A vertical BEP treatment system was also constructed at PKU to do experiments on the demountable cavity [12]. I-V curves at the buttons were measured and are shown in Fig.2. The measurements were done under a stationary condition where no acid movement was presented. We can see from Fig.2 that the current first starts to drop at the location near the iris, and then the midway. No sign of current dropping is seemed even if the voltage reaches the maximum output value of 30V. This observation implies that the film formation on the demountable cavity takes place first at the iris. With the bar shape cathode, it is possible that under a certain selected polishing voltage no uniform polishing can take place since the film only forms partially on the anode surface. This confirmed the earlier observation [1] that when the shape of cathode was modified to allow a more uniform electric field distribution inside an Nb dumbbell that was polished by BEP a shiny and uniform surface finish was obtained. Therefore selection of an optimized cathode shape can be very critical for BEP as discussed in the next topic.

More and better (less noisy) data are needed in order to be able to draw a conclusion from the I-V curves measurements on the button samples.

Analyses of the surface morphology and chemical composition of the buttons will be important in order to understand quantitatively how good a polishing process is for a particularly chosen set of polishing parameters. This work has yet to be done.

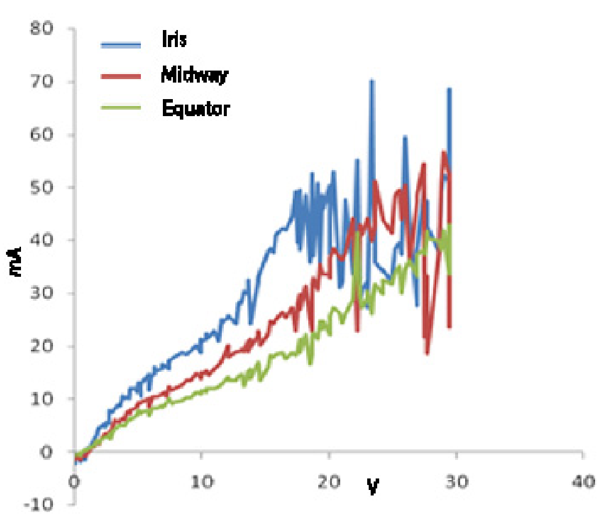

Figure 2: I-V curves of the buttons of the demountable cavity when a pure Al bar was used as the cathode [14].

CATHODE SHAPE

Experimental results shown in the last topic imply that cathode shape can be important during BEP treatments of Nb SRF single cell cavities. In fact, it was postulated in reference 13 that the cathode shape is important in general for EP of Nb. It was suggested [13] that Nb removal rate could be more homogenous if a shaped cathode that could produce more uniform electric field distribution was used in EP treatments of Nb SRF cavities.

For this study topic, three cathode shapes – bar, ellipsoid, and ball were fabricated as shown in Fig.3. Teflon tape was used to wrap up some parts of the cathodes so that minimum polishing would take place on beam pipe parts of an Nb SRF cavity. These three cathodes were used in the vertical setup that was built at JLab as described briefly in reference 2 and in more details in reference 5. For this study, the same polishing parameters were used for each cathode type for doing BEP treatments on CEBAF shaped cavities so that the only difference here was the cathode shape. Typical polishing parameters are: a) The flow rate of the electrolyte was significantly higher than 10 L/min (calibrated to water). b) The temperature of the electrolyte as measured from a thermometer mounted on the outer surface of the cavity at equator was kept below 35 ^{o}C. c) In this study, voltage control was adopted and was set at 18 V.

The surface finishes of the cavities treated by BEP using the three different cathode shapes differ significantly from one to the other. When the bar shape cathode was used, no polishing took place. The surface became matt and dark and seemed to be rougher than that before BEP. There were also some black strips on some locations of the inner surface. Surface finish was improved when the elliptical cathode was used. The area close to iris was clearly polished and was shinny and smooth. However, about one inch away from iris no polishing took place When the ball shape cathode was used, the polishing took place everywhere. The inner surface was visually shinny and smooth. However in the midway between the equator and iris a ring of pits was formed. The diameters of the pits range from submicron

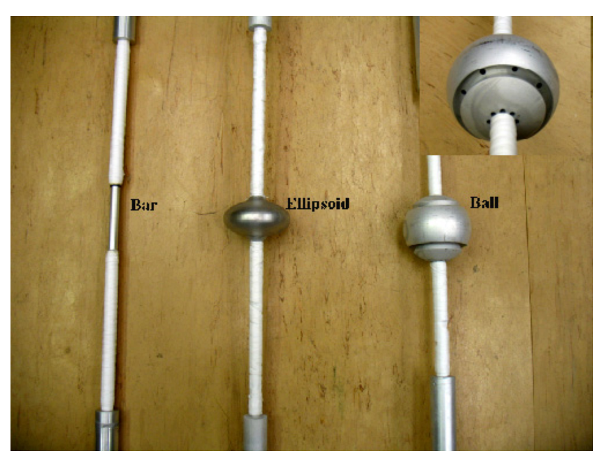

Figure 3: Three different shaped cathodes used in this study. The insert shows bottom view of the ball shape cathode.

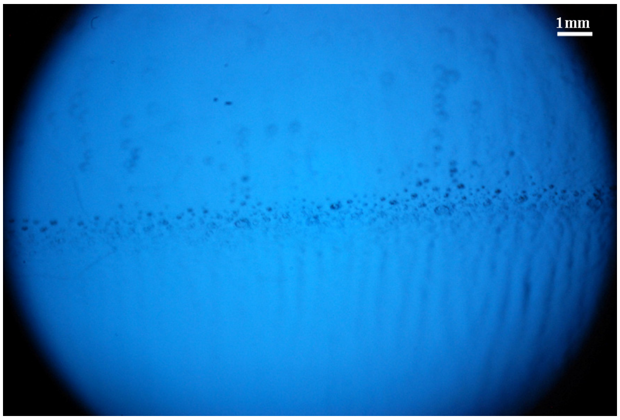

Figure 4: CCD image of the ring of pits located in the midway between iris and equator for the BEP treated Nb single cell cavity via the ball shape cathode (please read the text for details).

to a few microns as judging from an CCD camera (please see Fig.4). We believe that the pits were created since the polishing condition for this ring was near the gas evolution region of the I-V curve due partially to the short distance between the ring and the ball cathode and partially to a different electrolyte flow pattern at the ring.
Since the surface looked ugly for the cavity treated by the bar shape cathode, no RF test was done on the cavity. Fig.5 shows the RF test result on the cavity treated by the elliptical shape cathode. Q_o starts to decrease at about 9 MV/m, which is significantly lower than the on-set of Q drop for a normal fine grain cavity treated by buffered chemical polishing (BCP). Interestingly, Q_o is still reasonably high at 17 MV/m. It is not completely clear to us what is the mechanism responsible for the early Q degradation. Several cavities treated by BEP employing a commercially mixed BEP electrolyte showed severe Q-slope as observed on cavities treated both vertically and horizontally (please see reference 11). One obvious candidate is the matt and rough surface finish on the major part of treated surface. It was suggested [12] that some carbon precipitation might occur during BEP cavity treatments, which might be responsible for the degraded RF performance. Perhaps the cleaning procedure for the BEP treated cavities should be improved. We should not simply copy the successful cleaning procedure for EP treated cavities to BEP treated since the electrolytes are different between EP and BEP. For the cavity treated by the ball shape cathode with a ring of pits as shown in Fig.4, surprisingly the gradient reached 27.6 MV/m (quench limited) with an extremely flat Q as shown in Fig.6. The ring of pits did not seem to affect the RF performance very much. However the Q_o is very low. It was suspected that the low Q here could be caused by global heating. Therefore, thermometry measurements were done on this cavity after disassembly and cleaning. Fig.7 shows the result of the retest. This time the Q_o is reasonable and the gradient reached 25 MV/m as shown in Fig.7. The cavity was limited by quench with the

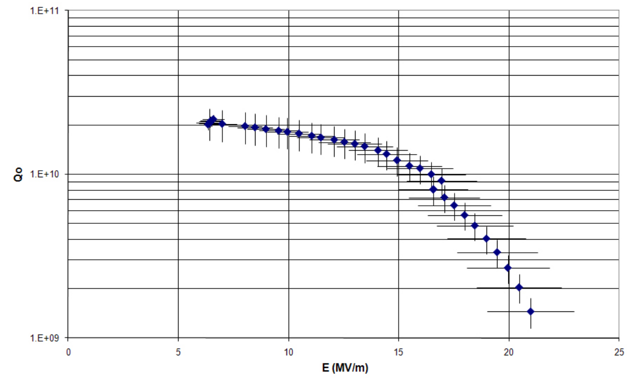

Figure 5: Excitation curve of the cavity treated by the elliptical shape cathode.

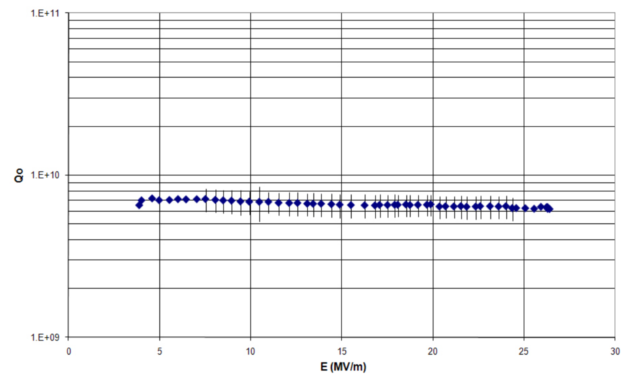

Figure 6: Excitation curve of the cavity treated by the ball shape cathode.

quenching site identified to be located close to the ring of the pits. Observations by an CCD camera on the quench site did not see any particular defects other than pits similar to those shown in Fig.4.

SURFACE MORPHOLOGY AND CHEMICAL COMPOSITION

It becomes clearer now that the surface morphology of the SRF cavities can affect RF performance significantly. Normally cavities with a smoother surface finish tend to

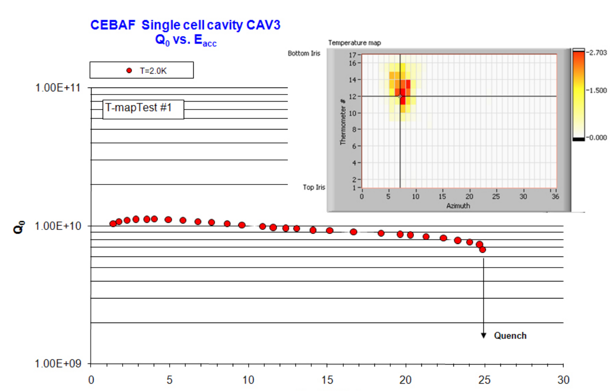

Figure 7: Second RF test result of the cavity treated by the ball shape cathode with thermometry mapping. The insert shows the quench site (please read the text for details).

show higher Q_o and have more chances to reach a higher acceleration gradient. Obviously, another important feature of the surface of an Nb SRF cavity is its chemical composition.

Several Nb flat samples were treated at PKU via the fast BEP technique [14] and then the samples were shipped to JLab for further surface characterization via profilometer, scanning electron microscope (SEM), energy dispersive x-ray (EDX), and static secondary ion mass spectrometry (SIMS) [15]. For details about the results of the measurements, please read reference 14. It was found that the smoothest surface could be obtained when the movement of the electrolyte with respect to the polished Nb anode surface reached a certain value [14]. Therefore the movement of electrolyte during BEP is very important. It can not only enable a much faster Nb removal but also create a smoother surface finish. The smoothest surface obtained on samples prepared by PKU is shown in Fig.8 where an RMS of 20 nm is measured by a high precision 3-D profilometer [15] over a surface area of 200X200 μm^2. To the best of our knowledge, this is the smoothest Nb surface ever reported in the literature. Surface feature similar to a finger print as first reported in reference 16 was also found on samples treated by PKU's fast BEP technique. At this moment, it is not completely clear how the pattern will affect the RF performance.

Six samples treated by EP and BEP were sent from CEA Saclay to Jlab for surface characterization. Dynamic SIMS measurements were done to the samples to see whether there are significant differences in terms of chemical compositions between samples treated with electrolytes either mixed in different ways or with different fluorine concentration. Typical results are shown in Fig.9. Not much difference was found between the two samples.

It is important to point out here that the SIMS results shown here may only be considered as preliminary since the samples were shipped to Jlab in plastic bags. Sample surfaces were in contact with organic materials, resulting strong hydrocarbon peaks and PDMS peaks that may overshadow contributions from other important elements. More measurements are needed to look for possible contributions from carbon and other elements as a result of the presence of lactic acid in the electrolyte.

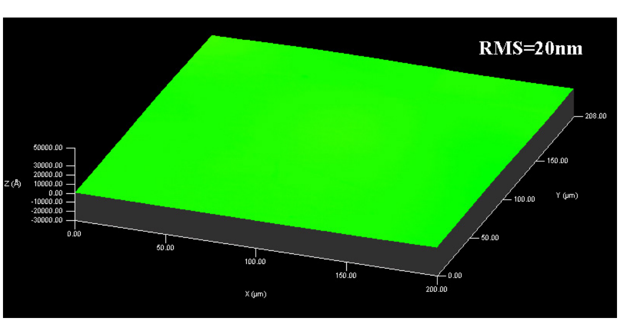

Figure 8: Profilometer image of an Nb sample prepared by PKU's fast BEP technique (please read reference 14).

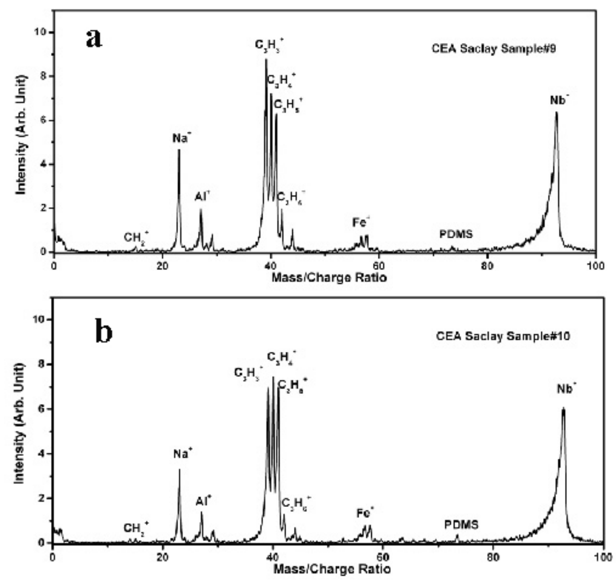

Figure 9: Dynamic SIMS measurements on CEA Saclay's sample#9 a) and sample#10 b). Sample#9 was treated by EP for an Nb removal of 150 μm followed by HF(48%) rinse. Sample#10 was treated by BEP with commercially mixed electrolyte for an Nb removal of 140 μm.

VERTICAL VS. HORIZONTAL

In the beginning of this project, we intended to treat the same type of cavities vertically at JLab and horizontally at CEA Saclay with the identical polishing parameters so that we could compare which way of treating Nb SRF cavities was better. Due to various reasons, this intension was not realized. Only three cavities were treated by horizontal setup at CEA Saclay and all of them had an early Q degradation as shown typically in Fig.10a). In

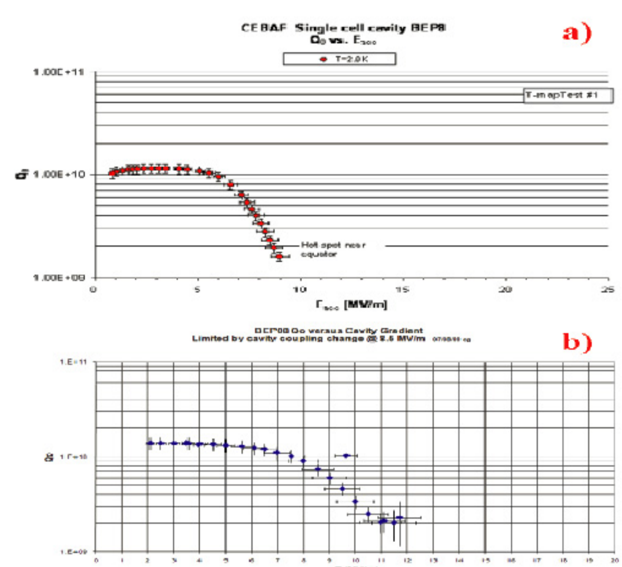

Figure 10: Typical RF test data on CEBAF cavity treated by BEP horizontally at CEA Saclay employing commercially mixed electrolyte: a) after the treated and cleaning, b) after BCP rinsed for two minutes and cleaning.

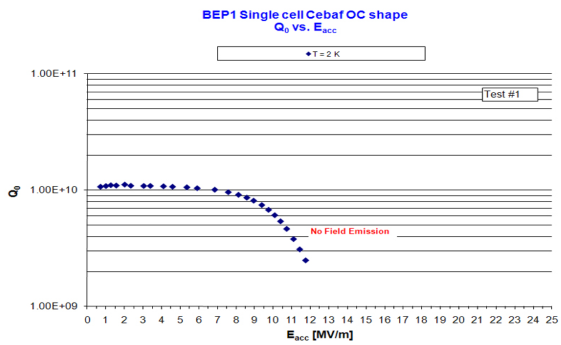

Figure 11: Typical RF test data on CEBAF cavity treated by BEP Vertically at JLab employing commercially mixed electrolyte.

this particular case, a hot spot was found near equator. This cavity was then BCP rinsed for 2 minutes and then tested again. No significant improvement was found as shown in Fig.10b).

In the early stage of this study, fine grain CEBAF cavities treated vertically using commercially mixed electrolyte shown similar RF results as those of Fig.10. A typical example is shown in Fig.11. Apart from variations in treatment parameters, the only other difference is the cathode shape between the cavities treated at CEA Saclay and JLab. For cavities treated at JLab, an elliptical cathode shape as shown in Fig.3 was used whereas at CEA Saclay a bar shape cathode was adopted.

After these discouraging results, a home-made BEP electrolyte was mixed at JLab and a ball shape cathode was fabricated. Inspired by the fast BEP technique developed at PKU, a much higher electrolyte flow rate was adopted to polish three CEBAF cavities vertically at JLab. All three cavities showed very encouraging results with accelerating gradients close to 25 MV/m and a decent Q_o. A typical excitation curve is shown in Fig.12.

CONCLUSION

In this paper, recent research progress on BEP is reviewed. Several important aspects of R&D on BEP are discussed in some detail. BEP as a new Nb surface treatment technique is still in its early stages of development. Based on the demonstrated abilities of BEP for achieving the smoothest surface finish on Nb and an an extremely high Nb removal rate of 4.67 µm/min, we anticipate that BEP can be a promising candidate for a next-generation surface treatment for Nb.

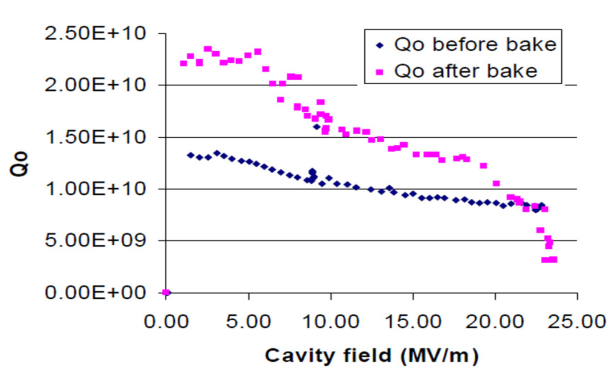

Fig.12: Typical RF test data before and after baking on CEBAF cavity treated by BEP Vertically at JLab employing home-mixed electrolyte and a ball shape cathode with high electrolyte flow rate.

ACKNOWLEDGMENTS

We would like to thank P. Kneisel for several cavities he made available to this project, RF testing, and much useful advice. Thanks Rongli Geng for taking the photos shown in Fig.4. Professional help from JLab's cavity production group is greatly appreciated. C. Crawford gave some help in the early stage of this project.

REFERENCES

[1] A.T. Wu et al, Applied Surface Science, 253 (2007) 3041
[2] A.T. Wu et al, Proc. of PAC 2009, Vancouver, Canada, (2009), in press
[3] X.Y. Lu et al, to be published
[4] F. Eozenou et al, https://indico.desy.de/conferenceDisplay.py?confId=1834, presented at TTC Meeting, Orsay, France, (2009)
[5] A.T. Wu et al, to be published
[6] B. Visentin, Proc. of the 11th Workshop on RF Superconductivity, Tuo01, Germany, (2003).
[7] J. Knobloch et al, Proc. of the 9th Workshop on RF Superconductivity, USA, (1999), p. 77.
[8] K. Saito, Proc. of the 11th Workshop on RF Superconductivity, Thp15, Germany, (2003).
[9] S. Jin et al, Proc. of the 13th Workshop on RF Superconductivity, WEP86, China,(2007)
[10] E. Wang et al, Proc. of the 13th Workshop on RF Superconductivity, WEP65, China, (2007).
[11] F. Eozenou et al, This proc. THPPO068 (2009)
[12] X.Y. Lu, Seminar at JLab, (2009)
[13] A.T. Wu, invited contribution to the book "Neural Computation and Particle Accelerators: Research, Technology and Applications", NOVA Science Publisher, New York, USA, (2009), in press
[14] Song Jin et al, This proc. THPPO097 (2009)
[15] A.T Wu, Proc. of the 11th Workshop on RF Superconductivity, Thp13, Germany, (2003).
[16] J.R. Delayen et al, Proc. of the 10th Workshop on RF Superconductivity, Japan, (2001) P499.