STUDY OF LOW TEMPERATURE BAKING EFFECT ON FIELD EMISSION ON NB SAMPLES TREATED BY BEP, EP, AND BCP*  

X.Y. Lu and K. Zhao, SRF Group, Institute of Heavy Ion Physics, School of Physics, Peking University, Beijing 100871, P. R. of China  
S. Jin, Thomas Jefferson National Accelerator Facility, 12000 Jefferson Avenue, Newport News, VA 23606, USA and SRF Group, Institute of Heavy Ion Physics, School of Physics, Peking University, Beijing 100871, P. R. of China  
L.C. Macintyre, Norfolk State University, 700 Park Avenue, Norfolk, VA 23504, USA  

Abstract  
Field emission is still one of the major obstacles facing Nb superconducting radio frequency (SRF) community for allowing Nb SRF cavities to reach routinely accelerating gradient of 35 MV/m that is required for the international linear collider. Nowadays, the well know low temperature backing at 120 °C for 48 hours is a common procedure used in the SRF community to improve the high field Q slope. However, some cavity production data have showed that the low temperature baking may induce field emission for cavities treated by EP. On the other hand, an earlier study of field emission on Nb flat samples treated by BCP showed an opposite conclusion. In this presentation, the preliminary measurements of Nb flat samples treated by BEP, EP, and BCP via our unique home-made scanning field emission microscope before and after the low temperature baking are reported. Some correlations between surface smoothness and the number of the observed field emitters were found. The observed experimental results can be understood, at least partially, by a simple model that involves the change of the thickness of the pent-oxide layer on Nb surfaces.  

INTRODUCTION  
Baking Nb superconducting radio frequency (SRF) cavities at 120 °C for 48 hours is a common procedure used in the SRF community to improve the well-known high field Q slope. Generally it is believed that heat treatments are beneficial [1] to the performance of Nb SRF cavities in terms of field emission since it can remove some physisorbed gases on the surface of Nb among other possible effects depending on the heat treatment temperature. This is thought to be consistent with some preliminary experiments [2] that we did on some small flat Nb samples via a unique home-made scanning field emission microscope (SFEM) [3]. However, some recent cavity tests at Jefferson Lab show that field emission is increased after the low temperature baking. This calls for a more detailed investigation on this topic. In this paper, we report on preliminary results of the measurements of field emission on Nb flat samples treated by buffered chemical polishing (BCP), electropolishing (EP), and buffered electropolishing (BEP) [4] before and after the low temperature baking and discuss the possible mechanism responsible for the observed experimental results.  

EXPERIMENTAL  
The Nb samples used in this study are the same Nb used for fabricating Nb SRF cavities at Jefferson Lab. All the samples were cut into a shape suitable for our SFEM experiments from the same Nb batch. These specially fabricated samples allow us to transfer the coordinates of the detected emitters from SFEM to the coordinates of our scanning electron microscope (SEM), energy dispersive x-ray analyzer (EDX), and electron backscattering diffraction (EBSD) system so that the emitters can be further characterized for their shapes, topographic information, chemical compositions, and possible crystallographic orientations. 150 µm was removed from the surfaces of all the samples. BCP was done using the standard 112 acid mixture. EP was done at 51 mA/cm², whereas BEP was done at 171 mA/cm² in the plateau area of the measured I-V curve when both the electrolytes were in the stationary condition. After the polishing, the samples were first cleaned by DI water for 10 minutes and were blown dry by a nitrogen gun. Afterward, IPA was used to clean the samples for another 10 minutes followed by DI water ultrasonic cleaning for 30 minutes. Finally DI water was used to clean the samples under a class 1000 clean room environment for 10 minutes before being blown dry via a nitrogen gun.  
The SFEM system used in this study is described in Ref.3. It uses the SEM chamber as a load-lock entrance for samples. Through appropriate marking, the coordinate of a sample can be transferred from the sample holder of SEM to that of SFEM, which allows an emitter to be checked at the same location before and after field emission scans.  
This SFEM has the largest scanning diameter of 25 mm in the world. The spatial resolution of the system is 2.5 µm with an electric field gradient up to 140 MV/m. The apparatus is housed in a 10⁻⁹ Torr vacuum chamber with an in-situ heat treatment ability. So the samples were first scanned by the SFEM and then transferred to the heat treatment chamber for treating at 120 °C for 48 hrs
without the exposure to air. After the treatment, samples were transferred to the measurement chamber for scanning.

The surface morphology of the samples was studied employing a P15 profilometer manufactured by KLA-Tencor. It has a vertical resolution as high as 8 nm and a guaranteed reproducibility of 0.75 nm. The scan size is 200X208 μm².

RESULTS AND DISCUSSIONS

We started this investigation in 2005. At that time, we were just curious about what the effect it had on field emission after the low temperature baking. The unpublished result of this initial investigation is shown in Fig.1. We can see from Fig.1 that the number of the detectable field emitters decreases from 104 to 67 after the baking. This appeared to fit into the pictured that the low temperature baking is beneficial to field emission since the gas absorption on Nb surfaces was removed by the baking that was generally believed [1] to reduce field emission. Then we thought that the issue was well understood and the study was done. However, recently several cavity tests at JLab show that field emission can be enhanced by the low temperature baking at 10⁻⁷ mbar. This triggered our desire to revisit the effect in a more systematic way.

Fig.2 shows a typical example of the measured results for a BCP treated sample. The number of emitters was found to increase slightly from 710 to 743. BEP treated sample showed a similar trend as shown in Table 1. Also included in Table 1 is the number of emitters detected on an EP treated sample. No data is available for the EP sample after the baking since the measurement is still going on at the time when this report is written.

The first thing that one can see from Table 1 is that the number of field emitters is significantly larger for the BCP treated sample. This implies that the number of foreign particles is significantly more on the BCP treated surface. One obvious reason for this is that the BCP treated surface is very rough and the applied cleaning procedure here is not good enough to remove all foreign particles. To check such a possibility, profilometer measurements were done on the three samples treated by BEP, EP, and BCP. The result is shown in Fig.3. The root mean square (RMS) of BCP surface is 996 nm that is significantly rougher than 472 nm of EP and 241 nm of BEP. This confirms again that a smoother Nb surface is desirable for good RF performance.

From the first row of Table 1, we can easily see that the number of detected emitters decreases from BCP to EP to BEP. This correlates with the results shown in Fig.3 very well, revealing that rougher surface generally tends.
to accumulate more foreign particles and thus is easy for field emission to take place.

How to understand the results presented in Fig.1 and the first and third columns in Table 1? We feel that this can be understood by the change of the thickness of the Nb\(_2\)O\(_5\) layer on the Nb surfaces before and after the low temperature baking. The measurement configuration of the SFEM can be viewed as a sharp metal tip on top of a wide metallic piece that has a thickness of 3 or 4 mm. The surface of this metallic piece is covered by a dielectric Nb\(_2\)O\(_5\) layer. Suppose the diameter of the SFEM tip is 200 µm and the gap between the oxide layer and the SFEM tip is 0.2 mm. When the applied voltage to the SFEM tip is 7 kV, the distribution of the electric potential can be shown in Fig.4. A simple simulation can then be done to see the variation of the electric field on the surface of the Nb pent-oxide surface as at the top of the tip as a function of the thickness of the pent-oxide layer. The result is shown in Fig.5. One can see from Fig.5 that the electric field decreases almost logarithmically with the thickness of the oxide layer. For instance, when the oxide thickness changes from 0.02 mm to 0.1 mm the electric field reduces by 0.5% only. So the results of columns 1 and 3 can be understood mainly as a result of thinner pent-oxide layers after the low temperature baking in vacuum. The reduced number of emitters observed in Fig.1 is a result of an exposure to air after the low-T baking due to a system break down. The exposure to air for a few hours might increase the thickness of the pent-oxide. Our results here indicate also that physisorbed gases on Nb surfaces do not play a major role in determining field emission characters of our samples. Further study is underway in order to understand this topic better.

REFERENCES