EFFECTS OF THE THICKNESS OF NIOBIUM SURFACE OXIDE LAYERS ON FIELD EMISSION*

A.T. Wu[1], S. Jin[1,2], X.Y Lu[2], R.A. Rimmer[1], and K. Zhao[2]

1: Institute for SRF Science and Technology, Thomas Jefferson National Accelerator Facility, Newport News, VA 23606, USA.

2: State Key Laboratory of Nuclear Physics and Technology, Institute of Heavy Ion Physics, School of Physics, Peking University, Beijing 100871, China.

Abstract

Field emission on the inner surfaces of niobium (Nb) superconducting radio frequency (SRF) cavities is still one of the major obstacles for reaching high accelerating gradients for SRF community. Our previous experimental results [1] seemed to imply that the threshold of field emission was related to the thickness of Nb surface oxide layers. In this contribution, a more detailed study on the influences of the surface oxide layers on the field emission on Nb surfaces will be reported. By anodization technique, the thickness of the surface pentoxide layer was artificially fabricated from 3nm up to 460nm. A home-made scanning field emission microscope (SFEM) was employed to perform the scans on the surfaces. Emitters were characterized using a scanning electron microscope together with an energy dispersive x-ray analyzer. The SFEM experimental results were analyzed in terms of surface morphology and oxide thickness of Nb samples and chemical composition and geographic shape of the emitters. The experimental results could be understood by a simple model calculation based on classic electromagnetic theory as shown in Ref.1. Possibly implications for Nb SRF cavity applications from this study will be discussed.

INTRODUCTION

The throughput of niobium (Nb) superconducting radio frequency (SRF) cavities has been always one of the important problems that SRF community has to resolve. This problem can become more serious when multi-cell cavities are produced. For instance, international linear collider will require 17,000 nine-cell Nb SRF cavities with an accelerating gradient $\geq$ 35 MV/m and $Q_o \geq$ 8X10^9. However, current RF test data of nine-cell cavities worldwide still scatter a lot. Many cavities cannot surpass the required gradient. One of the major reasons for this is that many nine-cell cavities suffer from field emission before reaching the gradient of 35 MV/m.

To seek the remedy for this problem, a study was launched two years ago on the effect of surface oxide layer on field emission. Preliminary results were reported in Ref.1. The study showed that the increase in the thickness of the Nb surface oxide layer appeared to be able to suppress the threshold of field emission. To understand this research topic better, a more systematic study has been carried out on a series of Nb samples with different thicknesses in their surface oxide layers obtained by anodization technique employing a unique home-made scanning field emission microscope (SFEM) [2]. This paper will report the results of this study.

EXPERIMENTAL

The Nb samples used in this study are the same as those used in Ref.1 and in cavity fabrication at Jefferson Lab. They were cut from the same Nb sheet and machined into a special shape that is suitable for scanning via the SFEM.

Eight specially shaped Nb samples were employed in this study. One sample was kept as the control. The others were subjected to anodization treatments in the mixture of ammonium hydroxide and de-ionized water at a volume ratio of 1:5. Different voltages from 25V to 230V were applied between anode and cathode as listed in Table 1. The obtained samples are shown in Fig.1. After the anodiztion, the samples were cleaned in exactly the same way as those done in Ref.1.

The SFEM system used in this study is described in Ref.2. It uses the scanning electron microscope (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 by SEM, energy dispersive X-ray (EDX) 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. In this study the high electric field reaches 94 MV/m.

Surface morphology of the samples was measured by a commercial profilometer P15 from KLA-Tencor. Vertical resolution of the system is 8 nm with a guaranteed reproducibility of 0.75 nm at a typical scanning area of 200X200 μm^2.

SEM and EDX measurements were done employing commercial Amary 1830 and Genesis Spectrum Version 5.21 by EDAX Inc.

All samples were intentionally exposed to class 1000 environment for 24 hours before being stored in clean

Table 1: Characteristics of the Anodized Samples

Sample	1	2	3	4	5	6	7
Voltage (V)	25	37	45	80	95	110	230
Color	Blue	Cyan	Bright Yellow	Green	Yellow	Red	Dark Grey
Oxide Thickness (nm)	53	77	93	163	193	223	463

07 Accelerator Technology

T07 Superconducting RF

Figure 1: Photo of the samples used in this study.

Petri dishes and later introduced into SFEM chamber.

RESULTS AND DISCUSSION

The results of SFEM scans on the control sample and sample# 7 are shown in Fig.2. 125 emitters were detected on the control sample whereas only 9 emitters were found on Sample# 7. To check whether the difference in the emitter number was caused by the difference in the surface morphology between the two samples, profilometer scans were done on the two samples. Typical profilometer photos are shown in Fig.3. The surface of sample# 7 appears to have more irregularity in small length scales. But interestingly the root mean squares (RMSs) of the two samples are very close. In fact, sample# 7 is a bit rougher than that of the control sample. This rules out the possibility that the morphology of the sample surfaces play an important role in this study. We do not expect that the chemical composition of the two surfaces should be different. EDX measurements on the two samples confirmed this expectation. Therefore, it is highly plausible that the difference was caused mainly by the difference in the thickness of the surface oxide layer.

Fig.4 shows a plot of measured number of emitters as a function of the thickness of the surface oxide layer for all samples including the control sample. It is important to note that the thickness of the surface pent-oxide layer of the control sample is 3 nm [3]. This natural oxide layer should be considered in the total thickness of the oxide layer in a sample as listed in Table 1 (it is known that 1 V of applied voltage corresponds to a growth of 2 nm in oxide layer during anodization).

It is clear that the number of the detected emitters does not decrease monotonically with the increase of the thickness of Nb surface oxide layer. However the general trend is that the samples with a thicker oxide layer have fewer numbers of emitters. The variation could be due to the random distribution of the airborne particles falling on
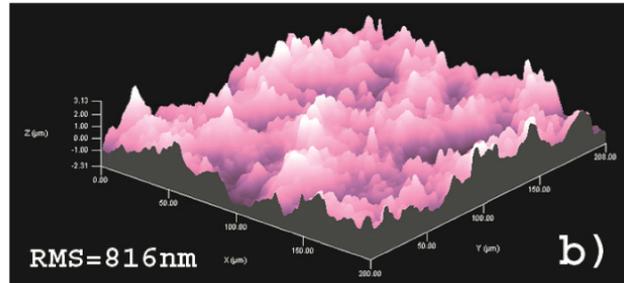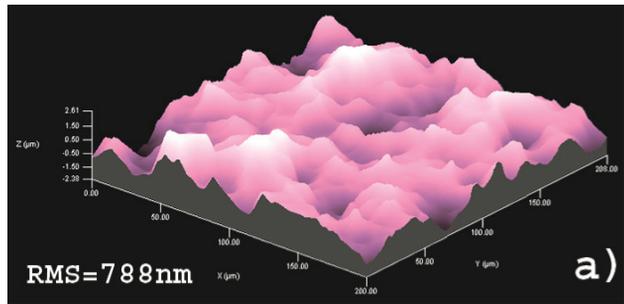

Figure 3: Typical results of profilometer scans on the control sample a) and sample# 7 b). The RMS of the measured surface is indicated.
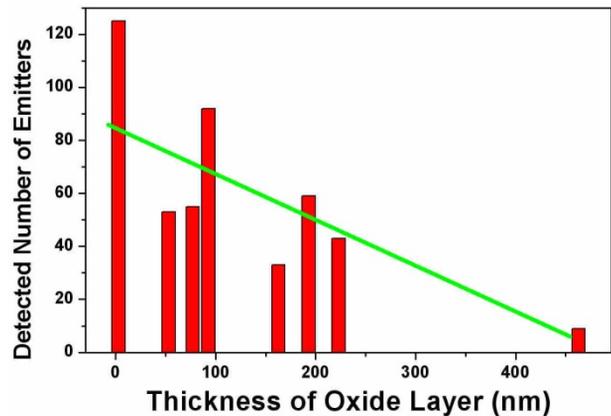

Figure 4: Detected number of emitters by SFEM as a function of the thickness of Nb surface oxide layer. The green line shows a guide to your eyes.

the sample surfaces during the exposure to Class 1000 environment.

The experimental results reported here seem to be consistent with what reported in Ref.1. It was demonstrated [1] through a simple model based on classic electromagnetic theory that the electric field on a Nb surface decreased when the thickness of the surface oxide layer increased. This implies that the onset field of a field emitter increases once the surface oxide layer becomes thicker. Therefore fewer field emitters will be observed on samples with thicker surface oxide layers. This is indeed what the trend of Fig.4 shows.

The detected onset fields of the emitters found on the surface of sample# 7 are shown in Fig.5. All of them are above 88 MV/m. This is exactly what predicted by the model calculation [1].

It is interesting to compare the on-set fields of the emitters found on sample# 7 with those found on the
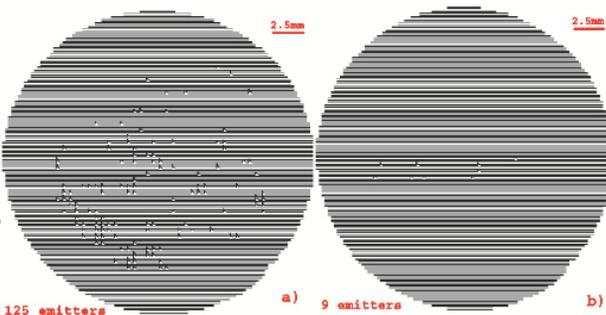

Figure 2: The results of SFEM scans on the control sample a) and sample# 7 b).

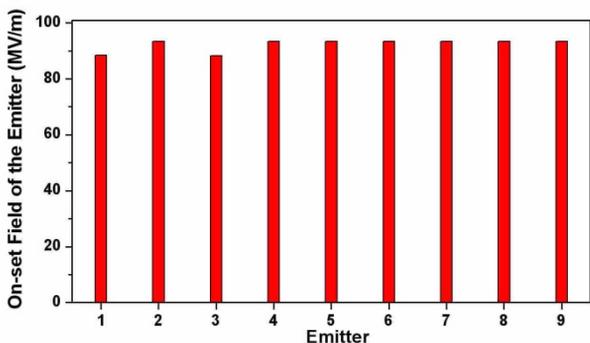**07 Accelerator Technology**

T07 Superconducting RF

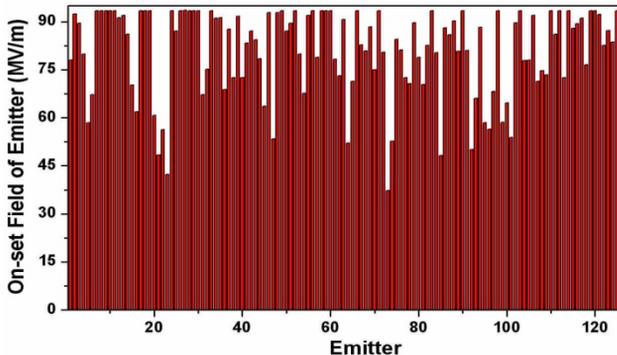Figure 5: On-set fields of emitters found on sample#7.

Figure 6: On-set fields of emitters found on the control sample.

control sample (see Fig.6). Surprisingly the on-set fields of the emitters on the control sample are not low at all. Only two emitters have an on-set field below 45 MV/m. At this moment, it is not completely clear whether all the detected emitters will cause field emission in a real cavity test. However, we know that if a Nb surface has fewer detectable emitters then the chance to get field emission from this surface is less.

We were curious about how the two emitters with an on-set field lower than 45 MV/m looked like in the control sample. By coordinate transformation between SFEM and SEM, it was possible to locate the two emitters. For the emitter with on-set field of 37.3 MV/m, we did not find an obvious emitter from SEM. But there was a black spot on the surface and several tungsten spots with sizes less than 1 μm as identified by EDX. It seemed that the emitter burnt or exploded during emission causing localized melting of the scanning tip. On the other one with on-set of 43.4 MV/m, Fig.7 shows the SEM photo of the emitter and its composition from EDX measurement.
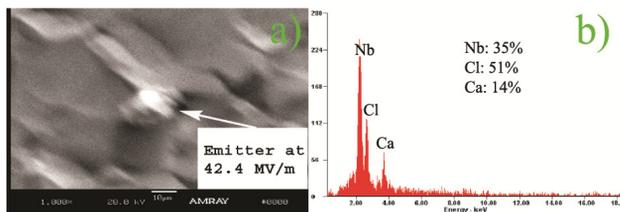

Figure 7: SEM image of the emitter with on-set field of 42.4 MV/m found on the control sample. b) is the EDX spectrum of the emitter showing it is composition.

Same measurements done on sample#7 showed that the detected emitters had relatively larger sizes and relatively smooth surfaces. A typical example is showed in Fig.8. It is plausible that some of them are airborne particles.
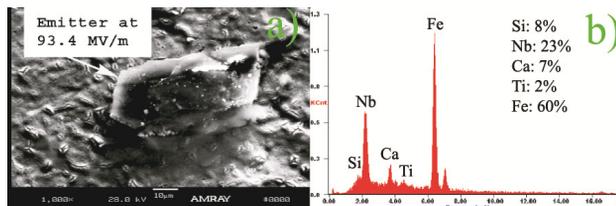

Figure 8: SEM image of the emitter with on-set field of 93.4 MV/m found on sample# 7. b) is the EDX spectrum of the emitter showing it is composition.

It is interesting to note here that the same surface can look differently under profilometer and SEM as one can see by comparing Fig.3 with Fig.7a) and Fig.8b).

How to apply the results from this study to Nb SRF cavity fabrication is not trivial. Our results seem to indicate that the thicker the surface pent-oxide layer the fewer the emitters will be on the surface. In order to find some suppression on field emission, we suggest that the thickness of the surface oxide layer has to be larger than 200 nm as judging from Fig.4. We expect that it is possible to suppress field emission completely if the surface pent-oxide layer is thick enough. Unfortunately, the electrolyte that we use for anodization processing here cannot allow us to go beyond 463 nm.

One uncertainty in this study is the sample exposure to Class 1000 environment. It is difficult to be completely confident that the observed differences in the numbers of the detected emitters are not caused by the random variation of airborne particles, although most emitters detected on the control samples at low on-set fields does not appear to be airborne particles as typically shown in Fig.7a). As the next step of this study, we plan to intentionally pollute each surface of the samples with equal amounts of Fe and Nb_2O_5 particles respectively as those done in Ref.4 to see whether we can still find a general trend in the detected numbers of emitters.

SUMMARY

The study reported here seems to indicate that a thicker surface pent-oxide layer on Nb surfaces is beneficial for suppressing field emission. More quantitative measurements are needed in order to verify this experimental result.

REFERENCES

[1] A.T. Wu et al, Proc. of IPAC 2010, Kyoto, Japan, WEPEC081
[2] A.T. Wu, Proc. of 11th Workshop on SRF, Germany, (2003), ThP13
[3] A.T. Wu et al, Proc. of 14th Conference on SRF, Germany, (2009), P300
[4] C.E. Reece et al, Proc. of 14th Conference on SRF, Germany, (2009), P746