

DC FIELD EMISSION STUDIES ON NB

T. Wang[†], C. Reece, R. Sundelin, TJNAF*, Newport News, VA 23606 USA

Abstract

The DC field emission studies in TJNAF aim to further explore the causes of field emission from Niobium surfaces and enhance our understanding of various preparation techniques for the purpose of improving field emission suppression. Field emitters on Nb samples are located by a DC field emission scanning apparatus in ultra high vacuum, then individually studied under SEM with EDS for characterization. Results at above 100 MV/m from samples prepared by BCP, and by electropolishing are presented and compared.

1 INTRODUCTION

During past years, the improvement of superconducting cavity preparation, dust free assembly and processing techniques have increased the acceleration gradient of Superconducting RF cavities to $E_{acc} \geq 20$ MV/m, but the reproducibility and further increase of gradient suffer from the breakdown of superconductivity induced by Enhanced Field Emission and quenching. Field emission limitation remains after the effective suppression of quenching by using Nb of high thermal conductivity.

DC field emission studies on Nb samples have been conducted in University of Wuppertal and Saclay since the pioneering work of Ph. Niedermann at University of Geneva. Field emitters are located and analyzed, and several models have been proposed for Enhanced Field Emission, including geometrical enhancement model, metal-insulator-vacuum (MIV) model, metal-insulator-metal (MIM) model, etc.

For the purpose of study, emission sources can be divided into four categories: geometrical damages (scratches, etc), external particles coming from handling or machining, impurities that are intrinsic to the material and possibly grain boundary due to its geometrical field enhancement. The former two categories, jointly referred as external emitters hereafter, are evidenced by past experimental results, while the latter two, referred as intrinsic emitters hereafter, haven't been directly confirmed or refuted for natural emitters.

Inspired by the recent interest in the superior results from electropolished cavities to BCP etched cavities, we examined and compared the field emission properties of BCP processed and electropolished samples, based on the different categories of emitters, in order to determine whether the difference, if exists, is intrinsic.

2 EXPERIMENTAL SETUP

The experimental apparatus, as shown in Fig. 1, is a UHV device, consisting of a SEM, a DC field emission

(FE) chamber and a heat treatment (HT) chamber attached together by gate valves. A magnetic coupling transporter can transfer a sample between these chambers in vacuum.

Within FE chamber, samples (slightly larger than 1" in diameter) can be moved in x, y, z by computer-controlled stepper motors, under anode tip for field emission scan (scan area: 25 mm dia.). The resolution in x, y, z movement is 2.5 μm . Anode tips are mounted on anode holder which can be moved linearly for tip exchange. The sample and sample holders in three chambers are specially designed for the sample to return to its previous location and orientation after non-in-situ processing or transferring between the chambers. Therefore, interesting spots can be relocated (with an accuracy of ~ 140 μm) for study. After emitters are located in FE chamber, sample is transferred to SEM chamber, equipped with Energy Dispersive Spectrometry (EDS) capable of window-less operation for light element sensitivity, for emitter characterization. Three artificial marks on sample surface are used to convert (x, y)s of FE chamber to (x, y)s of SEM chamber, and the usual accuracy in x, y relocation is ≤ 300 μm .

The gap between anode tip and sample is maintained by moving sample in x, y, z in coordination according to individual surface profile obtained by interpolating and extrapolating a 9-point profile data on surface. The gap consistency is estimated to be about ± 10 μm from a long distance optical microscope. The FE scan on entire surface is usually done using anode of 10 μm tip radius at a gap of 100 μm . The electrical field is dependent on anode shape, radius and gap and is calculated based on Ph. Niedermann's simulation and calculation [1]. After emitters are coarsely located, a local scan will follow around the emitter for finer location of the emission center at a gap of 50 μm . Gap can be calibrated by centering the anode at emission center and reducing the gap while adjusting the high voltage to output a constant current. The extrapolation to $V=0$ is set as gap=0. The field



Figure 1: Experimental apparatus.

[†]tong@jlab.org

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enhancement factor β and effective emitting area S can be obtained by linear fitting in order to characterize each individual emitter.

The HT chamber is designed for sample heat treatment by electron bombardment up to 1400°C, and the temperature can be monitored by an optical pyrometer.

The apparatus is located in class 100 cleanroom to reduce contaminants during sample handling.

The experimental circuit is illustrated in Fig. 2. The high voltage power supply is controlled by a PC to output a voltage ramp from 0 up to 30 kV, or until a current threshold, usually set at 1-2 nA, is reached, detected by a picoammeter. The scan process including stepper motor and instrument control, data acquisition and analysis are all performed by programs written in LabView.

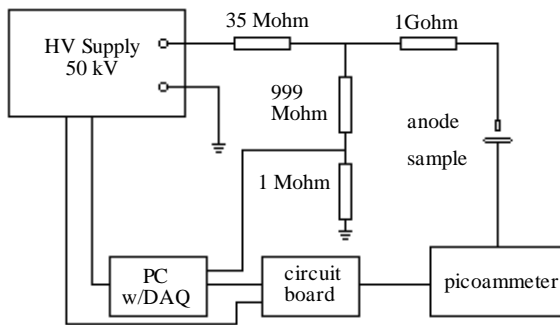


Figure 2: Experimental circuit.

3 RESULTS

3.1 Results from BCP Processed Sample

About 20 samples made of high RRR (~300) Nb are BCP (1:1:1) etched for various amounts of removal and scanned for field emitters at ~70 or 140 MV/m. Generally, four types of emitters are located besides emitters destroyed by vacuum arc: geometrical damages (scratches, etc), particles containing foreign elements, features with no foreign elements detectable by EDS, and emitters with no distinctive or resolvable features.

After some changes in sample handling to make sure that its surface is not touched by any means after sufficient amount of BCP removal, geometrical damages are reduced to zero. However, even with similar amount of BCP removal, emitter density still varies significantly from one sample to another, for example, total 8 emitters for the best sample, and over 60 emitters for the worst. In order to improve the reproducibility, machining process is examined first. A few rules, as listed below, are proposed and followed for new samples.

- inspect Nb sheet to choose defect-free material
- use a designated clean area for the machining
- use only plastic fixture
- change tools frequently as dull tool will embed impurity into sample

New samples are chemical etched, DI water rinsed in ultrasonic and then studied for field emission. The results at 140 MV/m are listed in Table 1.

Table 1: Results from new samples after BCP

	#65-test1	#65-test2	#65-test3	#63-test1	#63-test2
foreign particles	2	2	1	3	0
features-Nb only	0	3	0	1	1
emitters destroyed	1	2	2	4	4

*#65-test1, test2, test3 are after 250, 280 and 360 μm BCP removal respectively on sample #65. #63-test1, test2 are after 250 and 330 μm BCP removal respectively on sample #63.

Four of the foreign particles contain Fe, Cr and Ni, which could come from machining tools, and the rest contain Cu, Ca, Al, Ti; W, Ni, O; Fe; Ag; as well as Nb. They emit at a wide range of electric field, from 40 MV/m up to 140 MV/m. They are categorized as foreign particles because of their appearances and/or the fact that they are completely or partly washed away by further water rinse in ultrasonic. The sample edge, although not within the FE scan area, are found to have many machine damages and contaminants under SEM and EDS. While the origin of the particles is still unclear, further improvement in machining and particulate control will be pursued to reduce this category of emitters.

Emitters that appear bright under SEM but with no foreign elements detected by EDS are also found. The several micron profiling depth of EDS makes it unsuitable to detect very superficial elements, but with the setup of a new Auger system in our surface analysis lab, future analysis will be done for characterization. Nonetheless, these emitters only emit at ≥ 120 MV/m, which is significantly higher than normally achieved or currently desired surface field in cavities.

The last category of emitters are completely destroyed by vacuum arc, often occurred at lower or similar field for foreign particles, but EDS analysis indicated no foreign elements at arc sites. The mechanisms that initiated vacuum arc are very complex, and many theories for cathode-initiated arc are based on the initial heating of cathode emitter to reach thermal instability [2], which is unlikely for intrinsic emitter because of the good thermal contact with the bulk material. In the mean time, the transfer of weakly-bound microparticles from cathode to anode or vice versa, due to pure mechanical forces from strong electric field, has been proved by experiments [2] to start field emission and breakdown. Some microparticles caused vacuum arc but weren't completely destroyed by it and therefore can be studied and listed as foreign particles.

As shown in Table 1, emitter density is reasonably

repeatable from sample to sample, and further BCP removal doesn't monotonically reduce it, which indicates that the damage layer is already removed from the majority of the surface area and a fresh surface is revealed and studied each time. See Fig. 3 for a selection of emitter pictures.

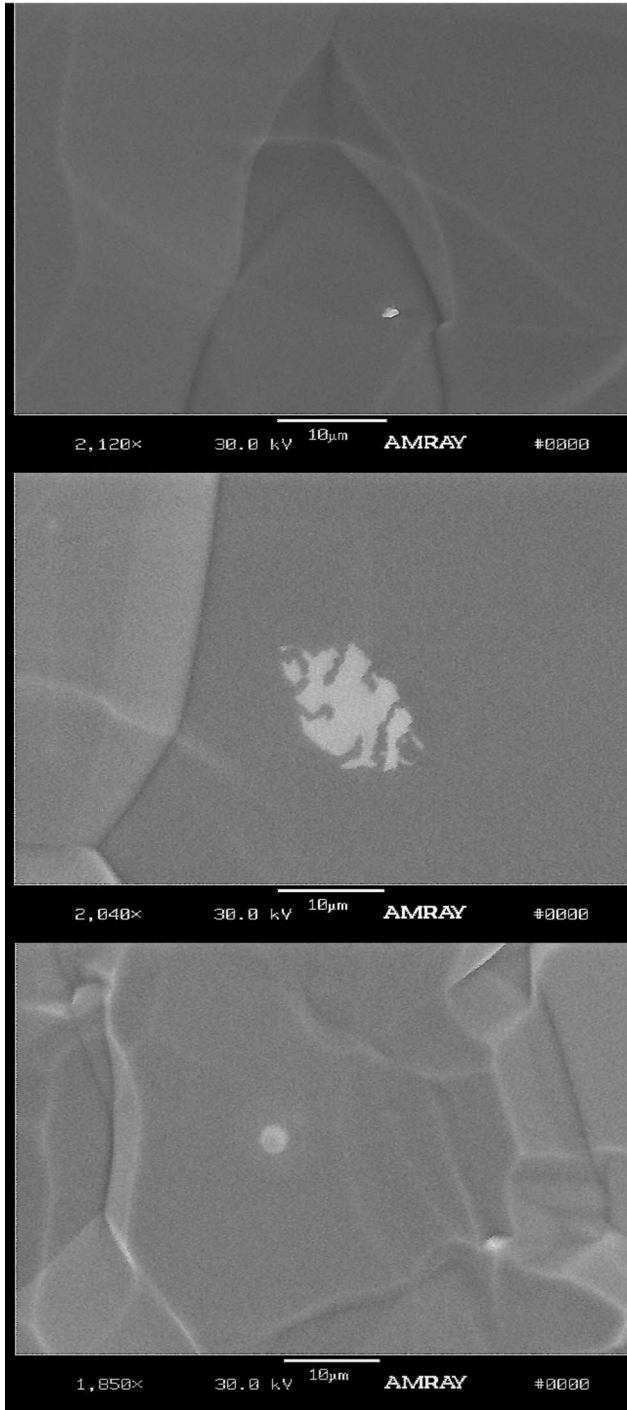


Figure 3: A selection of emitters on sample #65 and #63. top: foreign particle containing Nb, Fe, Cr, Ni, disappeared after another water rinse; middle and bottom: Nb only.

3.2 Results from Electropolished Samples

Sample #65 and #63 are BCP etched to remove 80 µm then electropolished to remove ~40 µm after above tests. The old "Siemens-recipe" (850 ml of sulfuric acid (96%) and 100 ml of hydrofluoric acid (40%)) is used for the electropolishing. The results are shown in Table 2.

Table 2: Results from electropolished samples

	#65 (440µm BCP+40µm EP)	#63 (410µm BCP+40µm EP)
foreign particles	1	0
features-Nb only	1	1
emitters destroyed	0	4

The one foreign particle, which caused a vacuum arc but wasn't destroyed by it, contains Nb, Fe, and Cr. See Fig. 4 for pictures of emitters with no foreign elements detected by EDS. One emitter site has six patterns with resembling details, two of which are shown in the bottom picture. Although their origin is unclear, they only emit at 100 MV/m or above.

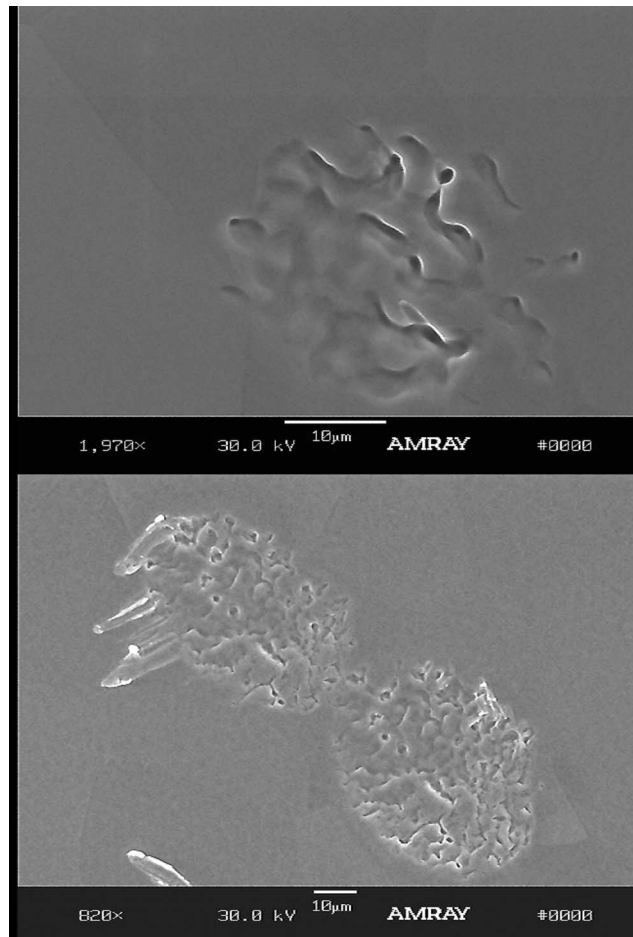


Figure 4: Emitters on electropolished sample #63 (top), on #65 (bottom), EDS shows Nb only.

4 DISCUSSION

From the results presented above, the following conclusions are drawn:

- No difference is observed in the field emission property of BCP and electropolished samples, at up to 140 MV/m.
- If assuming emitters destroyed by vacuum arc are microparticles, then no intrinsic emitters have been observed, at least up to 100 MV/m, for BCP or electropolished samples.
- Field emitter density is substantially reduced and consistent after improvement in machining process.
- More than half of the foreign particulate emitters contain Fe, Cr, Ni as well as Nb, which could come from machining tools.

More samples will be studied for better statistics. High pressure water rinse will be incorporated in the preparation process for further reduction in emitter density.

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6 REFERENCES

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