

# Enhanced Field Emission of Niobium Single Crystals

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## Abstract

In the last years several groups have shown that enhanced field emission (EFE) is the limiting factor to reach higher electrical field gradients in rf superconducting accelerator cavities. Therefore we study EFE from high purity niobium cathodes and characterize individual emitting sites with an UHV field emission scanning microscope. In this paper we present first field emission measurements on heat treated Nb single crystals of centimeter size.

## 1. INTRODUCTION

Enhanced field emission from broad area niobium cathodes is a subject of considerable interest at present because of its technological importance in high voltage vacuum devices and high field rf superconducting accelerator cavities [1]. The enhanced electron emission often originates at certain sites. The I-V characteristic of a typical field emitter (FE) generally follows a Fowler-Nordheim law which involves a field enhancement factor  $\beta$  and an apparent emitting surface area  $S$  [2]. In early investigations EFE was explained on the basis of a geometric field enhancement from sharp metallic protrusions on a cathode surface [3]. Nowadays, with the use of high resolution Secondary Electron Microscopy (SEM), it is observed that these metallic whisker are very rarely the cause of EFE. It is established that EFE most typically originates from localized pointlike microparticles on a broad area cathode surface. There are usually only a few active sites per  $\text{cm}^2$  which cause enhanced field emission [4]. Therefore it is essential to study individual emitting sites in order to control and reduce this field emission and to understand the under-

lying mechanism of EFE which is still basically unknown.

## 2. EXPERIMENTAL

The field emission measurements are done in a commercial UHV surface analysis system (VG Escalab) which is equipped with a Scanning Electron Microscope (SEM). The vacuum system also contains a separately pumped preparation chamber where the samples can be heated up to  $2000^\circ\text{C}$  by electron bombardment. A flexible sample transport system allows the transfer to the preparation chamber under UHV condition. For FE studies an UHV field emission scanning microscope (FESM) was built [5]. The FESM consists of a micromanipulator which performs precise cathode motions along the x-, y-, and z-axes. All three motions are driven by microprocessor controlled stepper motors. Seven anodes with different tip geometries for high and low resolution FE scans are mounted at a rotatable holder. A fast high voltage regulator allows FE measurements in a constant current mode [6].

From single crystalline niobium ( $\text{RRR}=800$ ) samples of 16 mm diameter and 3 mm thickness were machined into discs. For field emission measurements the cathode surface has to be very flat, smooth and chemically clean. To realize such well prepared samples we have chosen electropolishing with a standard recipe [7]. The investigated sample was then cleaned ultrasonically in alcohol and introduced into the vacuum system. In order to avoid a contamination of the surface the time between cleaning and the introduction into the system was very short. The field emission scans were done with a 0.3 mm diameter tungsten anode at constant gaps of  $140 \pm 7 \mu\text{m}$  and  $190 \pm 12 \mu\text{m}$ . During the scan the anode

was held at a fixed position while the sample was moved in a raster pattern. When a field emission site passes under the anode the applied high voltage is regulated to maintain a constant current of typically 5 nA.

### 3. RESULTS AND DISCUSSION

A selection of first field emission scans on a niobium sample which consists of two large single crystals is presented in Figure 1. These scans are for an as introduced cathode and after heat treatments (HT) at 820°C and 1400°C. After each thermal cycle the sample was systematically scanned at 57, 71, 85 and 100 MV/m with a low field emission current of 5 nA. From these scans it is evident that the density of sites drastically decreases after a HT for only 10 min at 1400°C and  $p=1 \cdot 10^{-6}$  mbar.

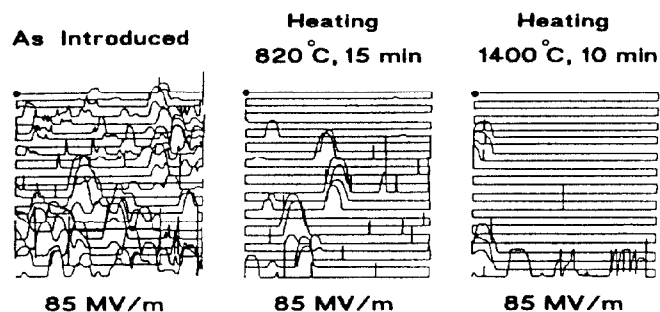


FIG.1 Low resolution FE scans,  $7.6 \times 7.6$  mm, of a high RRR Nb cathode, 0.3 mm diameter anode, limiting current 5 nA,  $d=140 \pm 7$   $\mu\text{m}$ .

In order to characterize the field emission sites with the field enhancement factor  $\beta$  and the emitting surface  $S$  we measured the Fowler-Nordheim characteristic of some selected field emitters. After heat treatment always a linear behaviour between  $\ln(1/E^2)$  and  $1/E$  was observed (Figure 2). Neglecting the image force correction in the Fowler-Nordheim analysis we obtained  $\beta=91-274$  and  $S=1.8 \cdot 10^{-9} - 6.0 \cdot 10^{-11}$   $\text{cm}^2$  which is in agreement with other measurements [8,9]. It should be mentioned that the real meaning of these parameters is still unclear because the FE current is almost certainly not coming from metallic protrusions on the surface.

For the annealed sample a single crystalline surface area of  $3.8 \times 3.8$  mm with no field emission

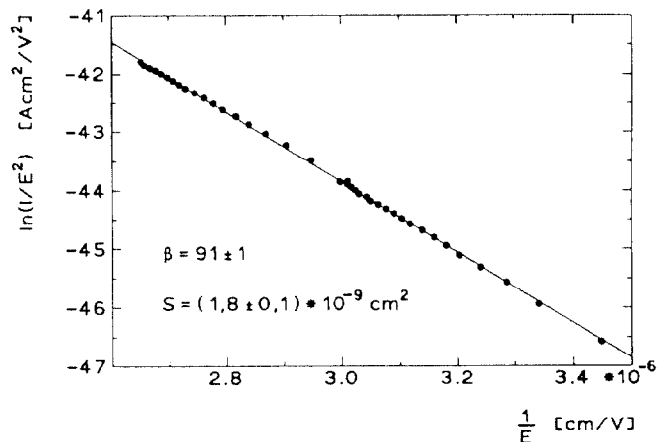


FIG.2 A typical Fowler-Nordheim plot, taken at a constant gap of  $d=140$   $\mu\text{m}$ .

activity at a current of 5 nA was measured. In order to check if a heat treatment above 1400°C creates new emitter on a single crystal we annealed the sample at 1700°C and 2000°C for 30 min in one thermal cycle. In the following investigations (Figure 3) no field emission was observed up to 111 MV/m. After this HT at 2000°C we observed that the sparking probability and the current noise was strongly increased at other parts of the sample. Even at low fields of 30 MV/m microdischarges and light emission were recognized. The following detailed investigations

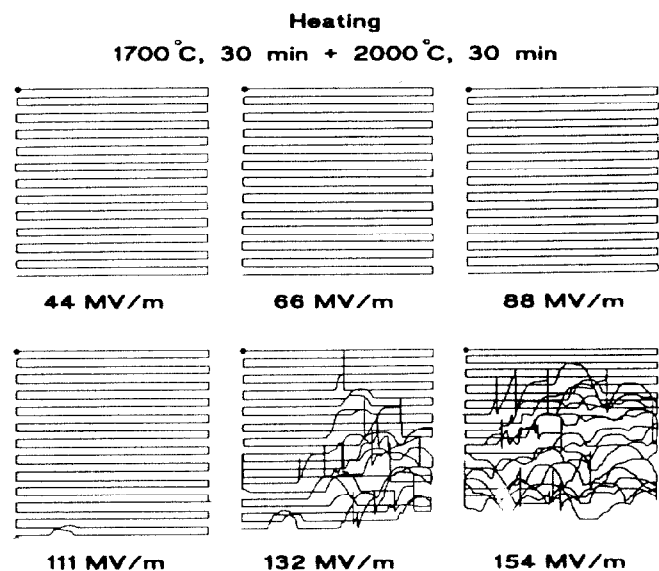


FIG.3 FE scans on a  $3.8 \times 3.8$  mm single crystalline area of the sample of FIG.1, 0.3 mm anode, 5 nA,  $d=190 \pm 12$   $\mu\text{m}$ .

of these sparking areas were done with a high resolution SEM. As a result we found about 20 splash marks [10] of molten niobium with a diameter of 50–200  $\mu\text{m}$ . Using Energy dispersive x-ray spectroscopy (EDS, sensitive to elements as light as Na) we found that all splash marks and craters (Figure 4) contained only niobium. In the surrounding area of some "special" molten craters only one additional foreign element, tungsten, was identified (Figure 5). We believe that each splash mark was produced after a microdischarge which was initiated by strong field emission activity. From the DC literature [11,12,13,14] it is known that explosive emission occurs as a consequence of Nottingham and Joule heating. In the first few ns after the explosion the total current density can reach  $10^8$ – $10^9$  A/cm<sup>2</sup> which results in a cathode erosion and

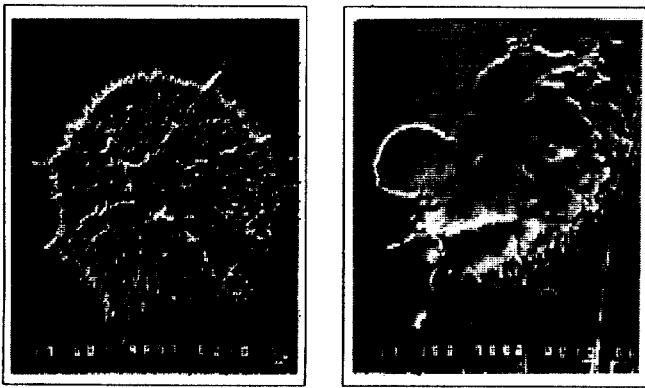


FIG. 4 SEM micrographs of the cathode after a DC field emission test in a constant current mode of 5 nA. Splash marks and craters of molten niobium (no foreign elements) are visible.

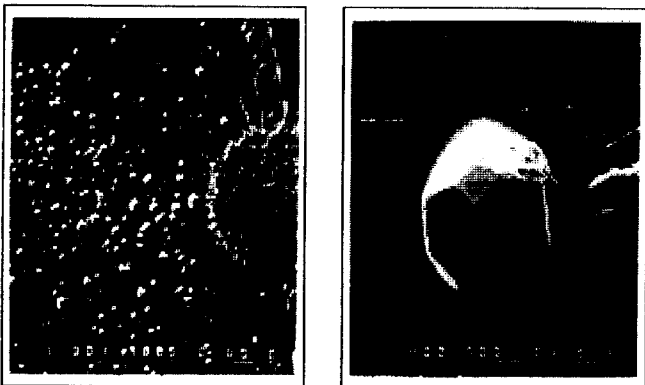


FIG. 5 SEM micrographs of a niobium splash mark surrounded by a field of small (2–10  $\mu\text{m}$ ) tungsten (probably from the anode) and niobium particles.

crater formation. In this process it is possible that a spray of the cathode (Nb) and/or the anode (W), caused by the vaporization of an emitter, can coat the sample surface with droplets.

#### 4. SUMMARY

We have measured the field emission properties of a single crystalline Nb cathode. After annealing at 820°C and 1400°C the density of field emitters decreased. On a Nb single crystal it was possible to obtain a nonemitting ( $I \leq 5$  nA) surface (3.8\*3.8 mm) at 111 MV/m after a HT at 2000°C. Microdischarges resulted in cathode and anode erosion with a crater and droplet formation.

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