

VERY LOW CURRENT FIELD ELECTRON EMISSION FROM ANODIZED NIOBIUM

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ABSTRACT

In this paper we present an experimental set-up for very low field electron emission DC current measurements ($I = 10^{-18}$ to 10^{-12} A) at room temperature. This device was used for studies on electropolished and subsequently anodized niobium samples. An interesting outcome is that the electron current is almost independent of the oxide layer thickness.

INTRODUCTION

One of the accelerating field limitations in niobium RF superconducting cavities is the field electron emission from the surface of the cavity [1]. As niobium exposed to atmosphere is always covered with a few nanometers thick niobium pentoxide (Nb_2O_5), we investigated the influence of the oxide layer on the electron emission. For this purpose we used anodized niobium samples of known oxide thickness. The experiments were performed in ultrahigh vacuum at room temperature. The electrode geometry was a planar niobium cathode opposite a grid. Behind the grid was an electron multiplier for the electron current measurement, allowing the detection of currents as small as 10^{-18} A.

EXPERIMENTAL SET UP (Fig. 1)

Because of the difficulty of field electron emission measurements inside RF superconducting cavities in cryostats we decided to study the emission at room temperature with DC field applied to the sample.

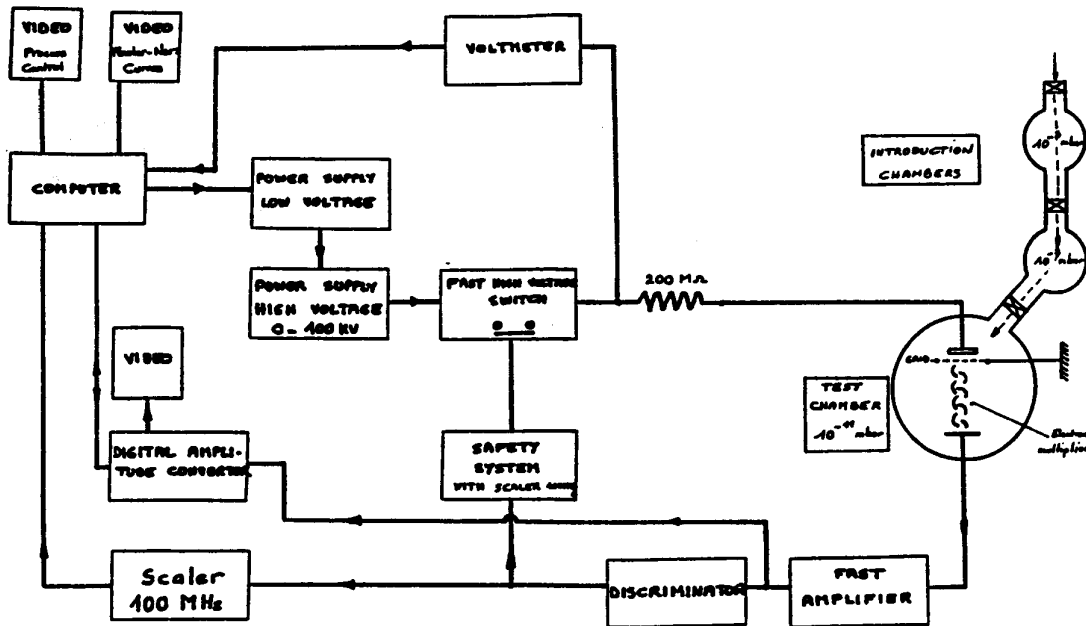


Figure 1 - Experimental set-up

The measuring gap is constituted by the sample in study as a cathode and a stainless steel grid as the anode. The electrode distance is of 1 mm. The grid is grounded, the negative high voltage is applied to the sample. The electrons emitted by the cathode pass through the grid and hit the first dynode of an electron multiplier.

The electrodes and the electron multiplier are in an ultrahigh vacuum vessel ($p = 10^{-9}$ Pa), evacuated by a 600 l/s ion pump and a titanium sublimator. Before the sample introduction the system is baked out at $T = 250^\circ\text{C}$ for several days. The sample introduction is performed through two intermediate chambers, the first one pumped up to 10^{-1} Pa by a primary membrane pump and a sorption pump, the second one pumped up to 10^{-5} Pa by turbomolecular pump. Thus it is not necessary to bake out the vessel after each sample introduction.

The current measurement is performed by the electron multiplier (gain 10^7) which collects the electrons and preamplifies the signal. This goes through a fast amplifier and a discriminator to a 100 MHz scaler that counts the pulses whose amplitude exceeds the electronic noise.

The experiment is computer operated. The computer delivers Fowler-Nordheim (FN) plots of the current-voltage characteristics : $\ln(I/E^2) = f(1/E)$, where E is the applied field and I the emission current.

SAMPLE PREPARATION

Anodic oxidation of niobium gives highly uniform and homogeneous films and requires little apparatus. A simple classical circuit including stainless steel cathode, DC power supply (400 V, 120 mA), and potentiometric recorder across the electrodes is available. In order to obtain accurate thickness values of oxide films, anodizations are performed by galvanostatic way at room temperature. When anodic oxidation is carried out at constant current density, the thickness W of the film is directly proportional to the voltage V supported by the cell. $dW = \alpha dV$. V is determined experimentally and the α values previously established [2] on niobium for various current densities. Our standard conditions for growing films (10 mA/cm² current density, in 0.1 N NaOH aqueous solution) lead to an α value of $20 \cdot 10^{-10}$ m/V. The electrolytes are prepared from double distilled water and reagent grade chemicals.

Before anodization the samples have been electropolished in the following way. After mechanical preparation of the surface : a 7 Volt DC voltage is delivered by a constant voltage power supply. The sample is settled as anode of the electrical circuit. The contact is made with a niobium wire soldered on the face opposite the one exposed to the electrolyte. An insulating paint is used to separate the solution from the contact face, delimiting a useful circular window on the face to be exposed. A large platinum electrode and powerful stirring are required. The process is generally limited to 10 minutes. The composition of the electrolyte is, for one liter :

H₂SO₄ (d = 1.84) 420 cc
Lactic acid (d = 1.23) 420 cc
HF 50% (d = 1.16) 160 cc.

These conditions lead to a particularly flat and specular surface.

RESULTS AND DISCUSSION

Different samples with a variety of oxide layer thicknesses were tested in the apparatus previously described. they all exhibit nearly the same curves that can be approximated by straight lines with similar slopes (within 10%) when plotted in the FN coordinates (Fig. 2 and Table I). So do the samples just electropolished and covered with a natural oxide film (Fig. 2 and Table I). This was observed for applied electric fields up to 25 MV/m and subsequent electronic currents up to 10^{-12} A and for a wide range of the oxide film thicknesses (20 to 350 nm). This shows that the electronic current is nearly independent

of the oxide film thickness. Such a result is contrary to that obtained by G.J.SAYAG et al. [3]. This is probably due to the quite different conditions in the experiments of these authors : larger applied electric fields, larger associated currents and conditioned electrodes.

Table I

| oxide thickness (nm) | FN slope ($\times 10^6$ A cm/V) | mean FN slope ($\times 10^6$ A cm/V) |
|-------------------------|----------------------------------------------------|------------------------------------------|
| natural oxide | -0.67 -0.73 -0.68 -0.66 | -0.69 |
| 21 | -0.61 -0.70 -0.69 -0.78 | -0.70 |
| 77 | -0.60 -0.68 -0.70 -0.67 | -0.66 |
| 147 | -0.67 -0.85 -0.99 -0.97 -0.85 -0.89 | -0.87 |
| 228 | -0.67 -0.70 -0.76 -0.73 | -0.72 |
| 247 | -0.84 | -0.84 |
| 285 | -0.84 -0.85 -0.81 -0.77 | -0.82 |
| 342 | -0.74 -0.81 -0.67 -0.52 -0.65 | -0.68 |

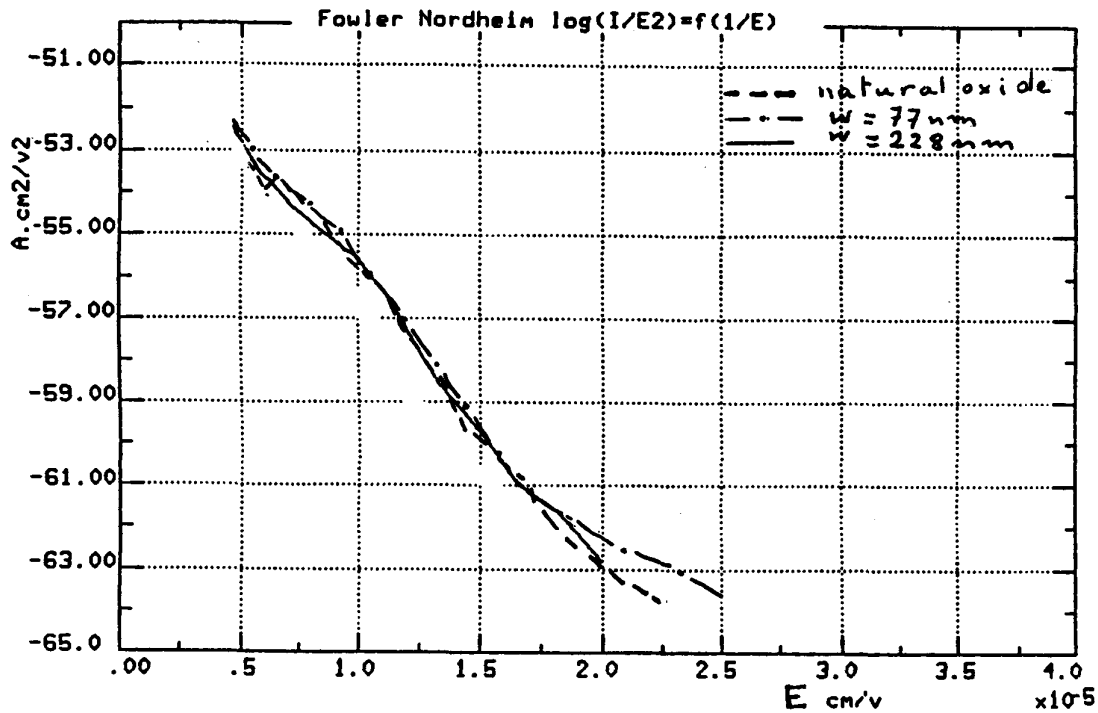


Fig. 2 - FN plots for anodized niobium samples

CONCLUSIONS

Earlier measurements of field electron currents were usually carried out using picoammeters. The minimum current is then of the order of $I_{\min} = 1.10 \cdot 10^{-12}$ A. In the experimental set-up presented here we used an electron counting technique for the measurement of the field electron emission current. This allowed us to obtain the very beginning of the electron emission curves ($I_{\min} = 1.10 \cdot 10^{-18}$ A). We found that the FN plots for these small values of the current are similar to those for larger ones.

The other outcoming result of our experiments is that for electropolished and subsequently anodized niobium samples the electron emission is almost independent of the thickness of the oxide layer.

REFERENCES

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