Investigating of the Field Emission Performance on Nano-Apex Carbon Fiber and Tungsten Tips

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Abstract: Field electron emission measurements have been performed on carbon-based and tungsten microemitters. Several samples of both types of emitters with different apex radii have been obtained employing electrolytic etching techniques using sodium hydroxide (NaOH) solution with different molarities depending on the material used. A suitable, home-built, field electron microscope (FEM) with 10 mm tip to screen separation distance was used to electrically characterize the electron emitters. Measurements were carried out under ultra high vacuum (UHV) conditions with base pressure of 10^-9 mbar. The current-voltage characteristics (I-V) presented as Fowler-Nordheim (FN) type plots, and field electron emission images have been recorded. In this work, initial comparison of the field electron emission performance of these micro and nano-emitters has been carried out, with the aim of obtaining a reliable, stable and long life powerful electron source. We compare the apex radii measured from the micrographs obtained from the SEM images to those extracted from the FN-type I-V plots for carbon fibers and tungsten tips.

1. Introduction

A high electric field applied to the surface of a specially prepared pointed metallic surface enables extraction of electrons from the metal by a quantum tunneling mechanism, which is called field electron emission (FE) [1]. On the practical side, the field emission can be used in the display devices by controlling the extracted electrons to hit a desired point on the tin-oxide conductive and phosphor-coated glass transparent screen [1]. Cold field electron emission (CFE) is an electron emission regime in which most electrons escape by tunneling from states below the emitter Fermi level [2]. With CFE, the characteristics of the emitted electrons are controlled by the width and height of the surface potential-energy (PE) barrier through which electrons tunnel [3]. CFE from the bulk is described by approximate equations known as Fowler-Nordheim-type (FN-type) equations [2].

The technical complete (generalized) F-N-type equation for the emission current $I$ can be written in the form [3-5]:

$$I = A \varphi^{-1} F^2 \exp\left[-\nu_F b \varphi^{3/2}/F\right]$$

where $F$ is the characteristic local barrier field, $\varphi$ is the local work-function of the base material, $a$ and $b$ are the first and second Fowler-Nordheim constants, and $\nu_F$ is the barrier form correction (which accounts...
for the particular shape of the potential barrier model), and $A_t$ is a parameter known as the formal emission area. Many kinds of materials such as carbon fibers and tungsten have showed great potential to be used in vacuum electronic devices such as, electron microscopes and other cold cathodes [6]. The attractiveness of particular types of field emitter is due to their favorable emission properties. The operating principle of such emitters is simple. In recent years, the interest in developing new types of field emitters as high brightness electron source for various technologically devices continues [7]. The most commonly used materials for such devices is tungsten [7-9]. In previous studies, Mousa et al. [10] and Baker et al. [11] found that carbon fibers can emit fairly stable currents in relative poor vacuum of lower than $1 \times 10^{-7}$ mbar. There have been serious studies to employ carbon fibers as electron sources in a variety of applications. This includes scanning tunneling microscopy (STM) [10,12], X-ray sources operated at high emission fluxes [13], and microfocus cathode ray tubes [10,14]. Inefficiency in having carbon fibers employed in electronic equipment is mainly due to stability problems. Forbes [10,15] illustrated this in a comprehensive review of the importance of carbon films and carbon-based materials in science and technological applications.

2. Experimental

Carbon fibers and tungsten emitters from micro and nano structures have been fabricated. For this purpose, tungsten wire with a diameter of 0.1 mm, and carbon-based fibers with a diameter of 7 μm were electrochemically etched in a 2 Molar and 0.1 Molar solutions of sodium hydroxide respectively [16]. After dipping about 2 mm from the sample in the solution, the etching process is started by using a power supply to provide the dc voltage until a certain initial current of about 30 μA for carbon fibers and 15 mA for tungsten is reached. Choosing a suitable etching current produces sharp tips at the liquid surface. After the etching process is completed, the sample is subjected to an ultrasonic cleaning process by using an ultrasonic device [17]. Following the cleaning process, the tip is mounted in a standard, home built, field emission microscope (FEM) with a tip screen distance of 10 mm [18,19]. The anode is formed as a conductive coated phosphor tin-oxide screen in order to allow for the recording of the emission current and the emission images [18,19]. The FEM was evacuated to achieve ultra high vacuum (UHV) condition, using two different systems incorporating a turbo molecular pumped system and an oil diffusion pumped system. The oil diffusion pumped system was connected to a liquid nitrogen (LN2) trap. Each of the two systems was connected with a rotary pump that reduced the pressure from atmosphere to $10^{-3}$ mbar. Afterwards, the turbo or the oil diffusion pump starts working and gave a base pressure of $10^{-7}$ mbar. To achieve the ultra high vacuum pressure ($10^{-9}$ mbar), the system was baked at 180 °C over night [17, 20].

To record the emission behavior in the vacuum, a high tension (EHT) power supply is used to apply negative voltage on the cathode (tip). The voltage is increased slowly until the emission current reaches about one microampere that is measured on a Kiethley 485 auto-ranging pico-ammeter. Then the applied voltage is slowly decreased until the emission current disappears. From the emission behavior of clean and sharp micro and nano emitters, a linear FN-type plot is expected [17,21].

3. Results and Discussion

The clean tungsten and carbon fiber microemitters presented in this work have been prepared with various apex radii to study and compare the properties of the field electron field emission from the respective materials. Selected results, including SEM micrographs of the tips' apex and the FN-type plots of the
electron field emission I-V characteristics, are presented in Figs 1-4. In this research work, a comparison was carried out between tungsten and carbon fiber apex radii measured from SEM micrographs and apex radii extracted from the FN-type plots as shown in Table 1. The relationship between the two methods will be discussed in the following sections.

3.1 Tungsten

SEM micrographs with high magnification (20 000X) (Figs. 1-4a) gives us the possibility to study the tips' apex shape profiles. The tip apex radius ($r_{SEM}$) has been derived from the graphically best fitted circle to the tip SEM micrograph. FN-type plots were used to derive the radii of the tips in more physical means as presented in Figs 1-4b. One has to analyze the I-V-type FN plots to obtain the plot slope, which afterwards will be the basis of deriving the second radius ($r_{FEM}$).

Many samples of both tungsten and carbon fibers were tested, with different apex radii. A selection of these samples is presented in this table. As it can be observed from ($r_{SEM}$) and ($r_{FEM}$) for the tungsten type, results showed usually that the ($r_{SEM}$) is less than ($r_{FEM}$) For carbon fibers the results obtained showed opposite trend, where the ($r_{SEM}$) is larger than ($r_{FEM}$).
Table 1

| Sample no. | Radius (SEM) $r_{SEM}$ (nm) | Radius (extracted) $r_{FEM}$ (nm) |
|------------|-----------------------------|----------------------------------|
| W 1        | 139                         | 183                              |
| W 2        | 220                         | 288                              |
| CF 1       | 90                          | 80                               |
| CF 2       | 115                         | 94                               |

The SEM micrograph presented in (Fig. 1a), shows clean and regular tungsten (W 1) tip apex shape. A radius of $r_{SEM,W1} = 139$ nm has been extracted. Due to the high regularity and symmetric status of the tip apex, there was no problem appeared in fitting the measuring circle to the tip apex.

From the FN-type plot (Fig. 1b), a typical linear behavior of the clean tungsten tip was obtained.

![Fig. 2. SEM micrograph (a) of a tungsten (W 2) tip at 20 000 times magnification. The FN-type plot (b) of this tip shows a typical behavior obtained from clean tungsten emitter, without any fluctuation during the voltage change. The slope of the FN-type plot was used to extract the tip radius ($r_{SEM}$). The extracted values are given in Table 1.](image)

No fluctuation, due to the voltage change, was observed. This meant that the field electron emission was obtained from a certain specific point on the tip apex, which enabled us to perform an accurate analysis. The slope of the plot was derived and analyzed, where the extracted radius is $r_{FEM,W1} = 183$ nm.

Fig. 2a presents a SEM micrograph for clean tungsten sample (W 2). The regularity and symmetrical shape and curvature of the tip were found. The radius of the tip apex was obtained, by fitting a circle on the SEM micrograph $r_{SEM,W2} = 220$ nm.

The FN-type plot for the same tip (Fig. 2b) produced a linear straight line which is consistent with the behavior of clean tungsten microemitters. Thus, the slope was obtained and analyzed, from which we extracted the radius of the tip apex $r_{FEM,W2} = 288$ nm.
3.2 Carbon Fibers

Using carbon fiber as an electron source, we faced more challenges towards the tip preparation and later on analysis of the results. Due to the small size of the fibers, it was hard to fix it into the stainless steel tube used to serve as the holding base for the fiber, as well as producing sharp tips through the etching process.

Fig. 3a shows a SEM micrograph of carbon fiber tip (CF 1). A non-uniform apex shape was obtained following the etching process. This might be as a result of leaving the tip in the etching solution for long time—less than 1 second—after the etch ended. Due to the non-ideal tip shape, we approximated the error $\Delta r_{SEM}$ as 20 nm. Applying the same techniques as in the case of tungsten, the tip emitting radius was obtained as $r_{SEM,CF1} = 90$ nm.

Fig. 3. SEM micrograph (a) of carbon fiber (CF 1) tip at 20 000 times magnification. The FN-type plot (b) of this tip demonstrates a typical behavior with a fluctuation effect which is probably resulted from the voltage change. The slope of the FN-type plot was used to extract the tip radius ($r_{FEM}$). The extracted values are given in Table 1.
The FN-type plot (Fig. 3b) shows a usual behavior of an ideal sharp tip of clean carbon fiber with small fluctuation. The fluctuation results from the applied voltage change and from the possibility of existing sub-emission centers on the emitting area. This could be observed in the emission images (not presented here). The slope was extracted and the radius was derived as $r_{\text{FEM,CF1}} = 80 \text{ nm}$.

The emission characteristics presented as a FN-type plot for the sample (CF 2) (cf. Fig. 4b) demonstrated the behavior of a clean carbon fiber. A small fluctuation was observed as a result of changing the applied voltage and might be from the irregularity in the curvature of the tip apex. The slope was extracted, and the radius was calculated $r_{\text{FEM,CF2}} = 94 \text{ nm}$. Comparison between both radii; namely $(r_{\text{SEM}})$ and $(r_{\text{FEM}})$ of clean tungsten and carbon fiber tips, is presented in (Table 1 and Fig. 5). On the one hand the tungsten emitter was found to fulfill and support the relation $r_{\text{FEM}} \approx 1.35 \times r_{\text{SEM}} \approx 20 \text{ nm}$ (see Fig. 5), which was discussed in previous studies [4,22,23]. On the other hand, the carbon fibers were not found to fulfill or follow this relation.

Fig. 4. SEM micrograph (a) of carbon fiber (CF 2) tip at 20,000 times magnification. The FN-type plot (b) of this tip shows a typical behavior with slight fluctuations’ effect which probably would be as a result from the voltage change. The slope of the FN-type plot was used to extract the tip radius ($r_{\text{FEM}}$). The extracted values are given in Table 1.
Fig. 5. Comparison between the values of $r_{SEM}$ and $r_{FEM}$ of clean tungsten and carbon fiber tips.

4. Conclusions

Electron micro and nano emitters from tungsten and carbon fibers have been compared in a systematic study. Using electrochemically etching techniques, similar to those employed by Müller in 1937 [24], emitters with different apex radii have been produced. Two completely different methods were used to determine the apex radii. The first was based on SEM micrographs, by graphically best-fitting circles on the SEM images to give $r_{SEM}$ directly. The second was based on FN-type plots, where the slope has been extracted, and the radius of the tips apex was calculated based on the extracted slope ($r_{FEM}$).

Tungsten based microemitters were found to fulfill the relationship obtained in this work which supports previous arguments [4,22,23]. The relation was used to relate the radii determined by two different methods namely SEM and FEM. Thus, the carbon fiber did not follow the same trend as tungsten. This might be because the tungsten tip apex usually had a more regular hemispherical shape. Thus the radius obtained from the SEM images of a tungsten tip is precise, and more directly comparable to the one extracted from the FEM. With the carbon fiber tips, due to the blurriness of the edges and the non-regular apex shape, it was more challenging to obtain precise apex-radius values from the SEM images. This made it hard to make a full and exact comparison with the radii extracted from the FEM measurements.

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