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Electron Microscopy-Based Performance Evaluation of Various Tungsten Field-Emitter Tips Apex Radii

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Abstract: Field electron emission measurements have been performed on emitters consisting of electrochemically etched tungsten (W) wires, where the microemitters apex radii have been varied ranging from 90 to 300 nm. A conventional field electron microscope (FEM) with 10 mm tip (cathode) - screen (anode) distance was used to electrically characterize the electron emitters under ultra high vacuum (UHV) conditions. By these means, the field electron emission characteristics; namely the recorded current with respect to the corresponding applied voltage (the current-voltage (I-V) characteristics) and the spatial current distribution have been recorded. Both, a scanning electron microscope (SEM) and a transmission electron microscope (TEM) were used to investigate the tips' profiles at high magnifications. Within this work, we compared and analyzed the data extracted from tungsten tips having different radii; and thus determined the deviations between the results of the two applied extraction methods. Mainly, we derived the apex radii of different tungsten tips by both electron microscopy methods and analyzed the I-V characteristics which are presented as the Fowler-Nordheim (FN) plots. The obtained results show a good agreement between the two methods (SEM and the FEM) that are used to extract the tip apex radii.

Keywords: Field electron emission; Field Electron Microscope (FEM); Fowler-Nordheim (FN) plots; Scanning Electron Microscope (SEM); Transmission Electron Microscope (TEM); Tungsten tips.

Introduction

Tungsten [1, 2] is still one of the materials which are most frequently used for field electron emitter tips manufacturing [3-5]. As a cathode material, it brings several benefits due to its favorable properties, such as: high melting point of 3422 °C (highest of all metals and second only to carbon among the elements), high level of hardness (strength), low vapor pressure, simplicity of emitter preparation bv electrochemical etching and durability as an emitter [1].

Within this work, various tungsten microemitters with different apex radii ranging from 90 - 300 nm were prepared. By using

electron microscopes ((SEM) and (TEM)) to extract the tip profile (i.e., apex radii) and by comparing the resulting radii with the radii extracted from the FN plots, one can calculate the deviation between both extraction methods.

The current-voltage (I-V) measurements were carried out (using FEM) under high vacuum conditions with a base pressure of about 10^{-9} mbar.

The remainder of this paper describes the emitter's preparation and the performed experimental work, followed by presenting the results obtained within this work and discussing them, while conclusions and future work are presented in the last section.

Experimental Setup

The cathodes incorporated here were electrolytically etched from 0.1 mm high purity (99.95%) tungsten wire (produced bv Goodfellow Metals Company) using a two-molar solution of sodium hydroxide. The etching process is controlled by choosing a suitable initial etching current of ~15 mA, where a voltage of 10-12V produced the required current for our experiments. After the etching process was accomplished, the etched sample was cleaned from any remains of the NaOH solution on the surface of the tip by being immersed in distilled water and subjected to an ultrasonic bath for some minutes. The prepared tip is then mounted in an ultra high vacuum (UHV) system, which is baked for 12 hours at a temperature of 180°C. The corresponding analyses were carried out using standard, home-built field electron а microscope (FEM) [6].

Before and after each of the previous steps, the sample was mounted on an optical microscope to obtain a first glance of the sample profile within small magnification (~1000X). Finally, the samples were mounted in TEM and SEM to obtain images at high resolution and magnification (up to ~30000X - 40000X).

Results and Discussion

The tungsten microemitters that were prepared have various apex radii ranging from 90 to 300 nm. The presented results include SEM [7-9] and TEM [10, 11] images of the emitters' apex as well as the I-V characteristics and FN-type plots of the field emission characteristics. The area efficiency α has been calculated as the ratio between the actual emission area required to generate the electron emission current (I) according to the FN theory and the area of the hemispheric emitter model given by $A = 2\pi r^2$.

The TEM and SEM images in Figs. (1-3) show smooth needle-like emitters, which possess approximately hemispherical tips. Fig. 1 presents a TEM image and an SEM image for sample W1 from which an apex radius of $r_{SEM,W1} = 145$ nm has been extracted. A small irregularity of the tip's apex was found. The I-V characteristics and the FN plots obtained for sample W1 during the

voltage decrease show a common emission current behavior and a linear FN plot of the clean tungsten microemitters [11].

The TEM and SEM micrographs are presented in Fig. 2 (a-b) for sample W2 from which an apex radius of $r_{SEM,W2} = 93$ nm has been extracted. A regular tip shape was found. The surface of the sample was contaminated most likely by the NaOH remaining on the surface as a result of insufficient sample cleaning. At the same time, it is shown that the apex of the tip was actually clean. The I-V characteristics and the FN plots obtained for the sample W2 during the corresponding currentvoltage cycle show a common emission current behavior and a linear FN plot as this is a characteristic for clean tungsten microemitters.

The emission characteristics obtained from a non-sharp tip with extracted apex radius of $r_{SEM,W3} = 215$ nm (cf. Fig. 3), with a uniform apex shape, show an interesting behavior. As the applied voltage is slowly increased through the virgin emitter, the emission current increases respectively while the voltage increases, at the low voltage region. At a higher voltage region, the emission current starts to behave in an unstable behavior (cf. Fig. 3 (c)). This clearly indicates that the emission at high voltage starts to emit from different spots (sub-emission centers) on the tip apex.

Fig. 4 (a-b) presents TEM and SEM micrographs of an exploded tip with an extracted apex radius of $r_{SEM,W3} = 100 \text{ nm}.$ The corresponding I-V characteristics were recorded before and after the apex explosion. As presented in Fig. 4 (c), before the apex explosion, the I-V characteristics behave as a sharp clean microemitter, where the emission current started at a voltage of ~ 500 V and increased respectively while the voltage increased. This shows a stable emission current, where the FN plot for the same sample (cf. Fig. 4 (d)) shows a general behavior as a linear plot.

After the apex explosion, the I-V characteristics and the corresponding FN plot (cf. Fig. 5) show a high similarity in the behavior similar to that before the apex explosion, only with a small difference that occurs in the emission-current starting voltage, where - after the apex explosion - the emission started from ~ 1100 V. This change occurs because of the fact that the emitting area became larger.



FIG. 1. Sample W1: (a) TEM micrograph and (b) SEM micrograph of a micropoint cathode at 30000X and 40000X magnifications, respectively, showing the tip apex shape and size, (c) plot that presents the corresponding I-V characteristics and (d) plot that presents the corresponding FN plot. Note that the FN plot is in a very good approximation that resembles a straight line which enables the calculation of the slope as $S_{W1} = 12715$.



FIG. 2. Sample W2: (a) TEM micrograph and (b) SEM micrograph of a micropoint cathode at 30000X and 40000X magnifications, respectively, showing the tip apex shape and size, (c) plot that presents the corresponding I-V characteristics and (d) plot that presents the corresponding FN plot. Note that the FN plot is in a very good approximation which resembles a straight line that enables the calculation of the slope as $S_{W2} = 4839$.



FIG. 3. Non-sharp tip sample W3: (a) TEM micrograph and (b) SEM micrograph of a micropoint cathode at 30000X and 40000X magnifications, respectively, showing the tip apex shape and size, (c) plot that presents the corresponding I-V characteristics and (d) plot that presents the corresponding FN plot. Note that the FN plot is in a very good approximation that resembles a straight line which enables the calculation of the slope as $S_{W3} = 23048$.



FIG. 4. Exploded tip W4: (a) TEM micrograph and (b) SEM micrograph of a micropoint cathode at 30000X and 40000X magnifications, respectively, showing the tip apex shape and size, (c) plot that presents the corresponding I-V characteristics and (d) plot that presents the corresponding FN plot. Note that the FN plot is in a very good approximation that resembles a straight line which enables the calculation of the slope as $S_{W4} = 5502$.



FIG. 5. Sample W4 after the tip apex explosion: (a) plot for the corresponding I-V characteristics and (b) plot presenting the corresponding FN plot. The slope is calculated as $S_{W4} = 9641$.

Fig. 6 shows the corresponding tested samples, at one magnification (1000X), using the utilized optical microscope.

The apex radius r_{SEM} for each tip has been derived from the corresponding SEM image by graphically fitting a circle to the tip apex. The blurriness of the edges was taken into account due to the irregularity in the apex shape as well as the contamination on the tip surface. The extracted apex radii r_{FEM} were derived from the FN plots and the relative emission area α has been calculated [12] from the extracted data. Table 1 shows the various obtained values of the apex radii utilizing both methods (SEM and FEM) and the corresponding relative emission area. The presented results do support the relation [13]: $r_{\text{FEM}} = 1.35 \text{ x } r_{\text{SEM}} - 20 \text{ nm}$, where Fig. 7 shows also the good agreement between the two extracted SEM and FEM tips apex radii.

It is worth noting that the investigated field emission is used to characterize various emitting materials in the continuous efforts to develop new types of electron sources and to understand the physics behind their behavior [14-18].



FIG. 6. Optical images at 1000X magnification: (a) W1, (b) W2, (c) W3 and (d) W4.

TABLE 1. Data of tungsten micropoints cathodes. The microemitters with various apex radii ranging from 90 to 300 nm were tested using SEM and FEM. The values of the apex radii were derived by the two methods (SEM and FEM) and showed close values with small deviation. The corresponding relative emission area (α) was also calculated.

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Sample no.	Radius (SEM)	Radius (extracted)	Relative emission
	r _{SEM} [nm]	r _{FEM} [nm]	area (extracted) α
W1	145	187	6.94×10^{-4}
W2	93	108	1.16×10^{-4}
W3	215	278	2.05×10^{-3}
W4	100	115	1.69×10^{-4}
W5	191	241	1.30×10^{-3}
W6	220	288	2.21×10^{-3}
W7	139	183	5.59×10^{-4}



FIG. 7. Comparing the values of r_{FEM} and r_{SEM} produced good agreement between the two extracted tips apex radii.

Conclusions and Future Work

This work introduces the calculations of the tips apex radii using two different experimental methods. The first method is carried out using graphically best-fitting circles on the SEM images (r_{SEM}), while the second one is performed by extracting the radii from the emission characteristics recorded in the FEM (r_{FEM}).

From the obtained results, one notes a good agreement for the values of the apex radii that are extracted by the two methods. Also, by comparing the extracted data with the previously recorded results, a high level of agreement with the introduced results is found. This indicates, as one important factor, that there was no relevant contamination by NaOH from the etching process on the tips' surface. The correlation within the data obtained through this work supports the corresponding relation between (r_{FEM}) and (r_{SEM}) as $r_{FEM} = 1.35 \text{ x } r_{SEM} - 20 \text{ nm}$.

Future work will include coating the samples with dielectric layers of epoxy resin and re-doing the performed investigations in new experimental setups to study the effects of coating on the resulted emission current behaviors.

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