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ARTICLE

Field Electron Emission Characteristics of Tungsten–Polyethylene Composite Material As a Source of Electron Emission

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Abstract: This work provides an experimental study on the effects of polyethylene coating on the field electron emission characteristics of clean (uncoated) tungsten tips. Several tungsten tips, with different apex radii, have been prepared, coated with different thicknesses of polyethylene layers and then examined using a standard field electron emission microscope. Various field electron emission characteristics have been measured under high-vacuum conditions. These include current-voltage characteristics, Fowler-Nordheim plots, scanning electron micrographs and spatial current distributions (electron-emission images). Based on this work, it is proved that coating tungsten tips with polyethylene layers has caused dramatic improvements on the tip emission properties. In particular, coating the tips improves the current-voltage characteristics, which is reflected in lowering the extraction voltage, getting more stable emission currents, expressing the current-voltage characteristics using Fowler-Nordheim plots and finally, the spatial distributions of the emitted electrons for the coated tips are more stable and uniform.

Keywords: Field electron emitter, Polyethylene dielectric layer, Composite emitter, Field electron microscope, Scanning electron microscope.

1. Introduction

Cold-field electron emission (CFE) is the emission of electrons from the surface of a condensed phase into another phase, usually vacuum, in situations where emission is induced by a high external electrostatic field [1-5]. It is a fundamental process in the operation of many technical applications, such as thermionic valves, cathode ray tubes (CRTs), X-rays and scanning and tunneling electron microscopes [6-8].

Many significant experimental studies of field emission have been carried out on carbon fibers, polymer graphite flakes and different tungsten and tungsten oxide nano-wires, such as (W, W_5O_{14} and $W_{18}O_{49}$) [9-13]. Advances in vacuum technology have developed the production techniques of field-emission cathodes, where high vacuum is essential for ensuring reliable operation of field-emission cathodes [10-17].

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The objective of this work is to study the alterations that may occur to the properties of field electron emission from various pure tungsten emitters [18] before and after being coated with different thicknesses of polyethylene layers. For this purpose, several tungsten tips with various apex radii have been prepared using electrochemical-etching techniques [19].

The current experimental study reports the current-voltage $I_{\mathrm{m}}(V_{\mathrm{m}})$ characteristics of the prepared tungsten emitters, Fowler-Nordheim (FN) plots and the scanning electron microscope micrographs (SEM) for measuring the thicknesses of polyethylene coating layers. Moreover, the emission-current distributions obtained from the different tips were photographed using phosphorescent screen [19-21].

The results of this research are divided into two parts; the first part includes the results from the uncoated tungsten tips, while the second part includes the results from the tungsten-polyethylene composite.

Using FN plots, the uncoated samples were tested using Forbes field-emission orthodoxy test [22] and the characterization parameters for the tested tips are extracted; mainly, the characteristic voltage-conversion length ζ_{C} and the related field-enhancement factor γ_{C}, in addition to the formal emission area from a Schottky-Nordheim barrier $A_{\mathrm{f}}^{\mathrm{SN}}$ [23]. This is considered to make sure that the used uncoated emitters are sufficient to be used in the second part of the experiment.

This is important, because these parameters can be used for monitoring the changes in the system cathode-anode geometry, which need to be evaluated in an orthodox setup that passes the field-emission orthodoxy test. So, the results from the uncoated tips can be compared to the results from the composite tips, because the changes in the system geometry can be neglected [22].

The tips' coating material (polyethylene (PE)) consists of non-polar, saturated, high-molecular weight hydrocarbons. The individual macro-molecules are dielectrics, because of their symmetric molecular structure and they tend to crystallize [21-22]. PE melting point is typically in the range of 120 °C to 180 °C. It has the chemical formula $(\mathrm{C_2H_4})_{\mathrm{n}}$ and its chemical structure is shown in Fig. 1. PE is usually a mixture of similar polymers of ethylene with various values of n [21].

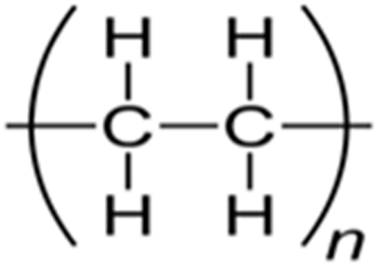

FIG. 1. Chemical structure of polyethylene.

2. Experimental Method

Tungsten micro-emitters have favorable properties, such as a high melting point of 3377 ˚C, a work function of 4.55 eV, high hardness (strength) and heat resistance at high temperatures [1]. Therefore, they are extensively used as electron emitters.

Tungsten micro-emitters typically have radii less than 100 nm and are produced by electrochemical-etching techniques [2]. The emitters were prepared by etching tungsten wires (~ 0.1 mm in diameter) using a 2M solution of sodium hydroxide (NaOH). The tip (anode) and a graphite rod (cathode) were connected to a power supply that provided the bias voltage necessary to generate the etching current. In this study, the bias voltage ranged from 10 - 12 volts. A multi-meter was used to monitor the current between the anode and the cathode; in order to quickly switch off the voltage when the etching process is complete, as presented in Fig. 2.

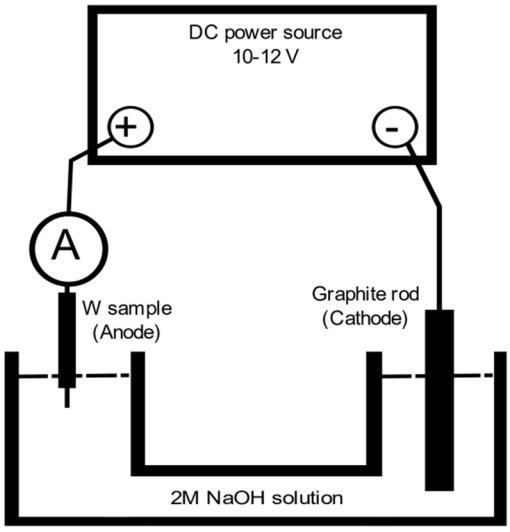

FIG. 2. Schematic diagram for the etching device.

The latter point is extremely important, as the cut-off time for the etching circuit greatly affects the sharpness and the shape of the tip [19]. The etched samples were then cleaned from any traces of NaOH solution by immersing them in distilled water and subjecting them to an ultrasonic bath for about 6-9 minutes [1].

To prepare the coating material, 0.5 gram of pure polyethylene was dissolved in 75 ml of xylene and the mixture was heated to 200 °C, where it became an almost 0.24 M-concentration solution. It should be noted that the coating process must be performed before the solution cools down.

Coating the tips with polyethylene involves two steps. Firstly, the tip of the emitter is dipped very slowly into the solution using a sample holder that keeps the sample vertical to the surface of the solution. The sample holder is attached to a trolley that can move the sample vertically to immerse the tip into the coating solution. Secondly, the immersed tip remains in the solution for five minutes at room temperature, so that the solution will cool down on the tungsten tip and the solvent (xylene) will vaporize, leaving the polyethylene solute stuck to the emitter surface.

The coated emitter is then mounted to a field electron emission microscope (FEM) system with an emitter to screen distance of 10 mm [1]. The screen images of the FEM are photographed directly using a digital camera.

The emission currents are obtained at a relatively high vacuum (~ 10–7 mbar). The high vacuum is maintained using an oil diffusion pump system. This pressure is obtained by baking the ultra-high vacuum system to ~ 160 °C for 7 hours and providing the system with liquid nitrogen (L-N_2) just after the baking process is completed [2, 22]. The radii of tip apexes were estimated through SEM; they were obtained at 20 kV, with magnifications up to ~ 6000x.

3. Results

SEMs of the prepared tips, before and after coating, with magnifications 800x and 6000x are presented in Fig. 3. The radii of the clean (uncoated) and coated tips were found to be 85.8 nm and 112.7 nm, respectively. Therefore, the thickness of the coating layer is estimated to be 26.9 nm.

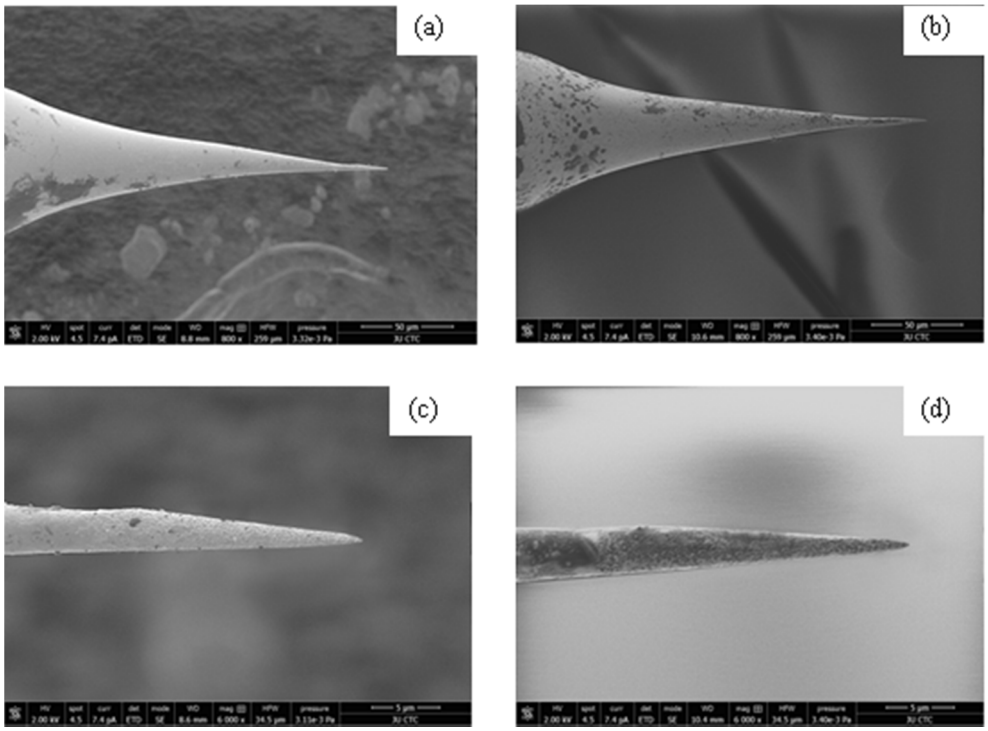

FIG. 3. SEM images for: (a) Uncoated sample at magnification (800X), (b) Coated sample at magnification (800X), (c) Uncoated sample at magnification (6000X) and (d) Coated sample at magnification (6000X).

In what follows, the experimental findings for both uncoated and coated (composite tungsten-polyethylene) tips are presented.

3.1 Characteristics of Uncoated Tungsten Tips

The $I_m(V_m)$ characteristics of the uncoated tips were recorded and presented in Fig. 4 for a full increase and decrease cycle of the applied voltage. The emission process started at 490 V with an emission current of 1 pA. The voltage was then raised slowly (to avoid the loss of the tip by any possible explosion) to 1000 V, which resulted in a current value of 820 nA. The voltage range is then decreased slowly until the emission current reached 9.1 pA at 520 V.

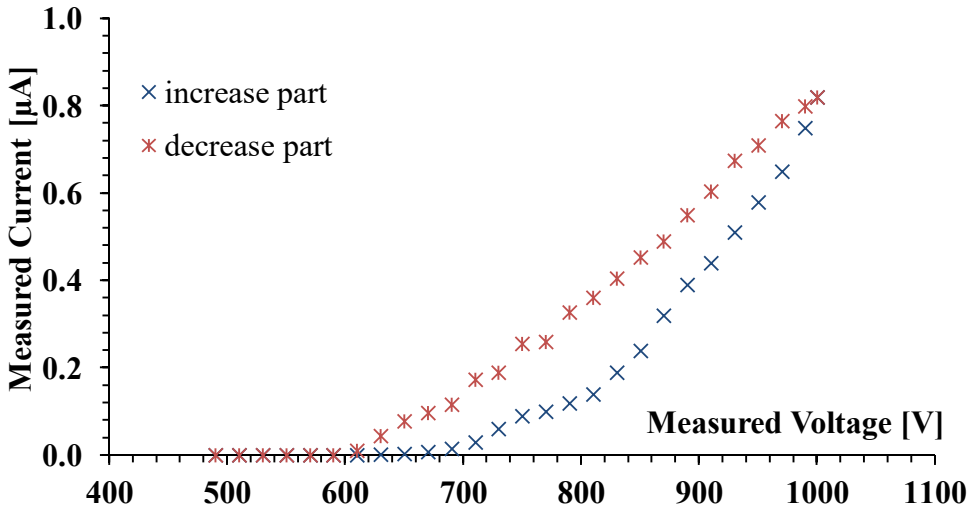

FIG. 4. The current-voltage characteristics for a full testing cycle of the applied voltage for the uncoated tip.

The related FN plots of Fig. 4 are presented in Fig. 5. From these figures, the field-emission orthodoxy test is applied for the voltage range 550-610 V of the increase part of the cycle; this part passes the test and the provided values of the characterization parameters for the uncoated emitter are presented in Table 1.

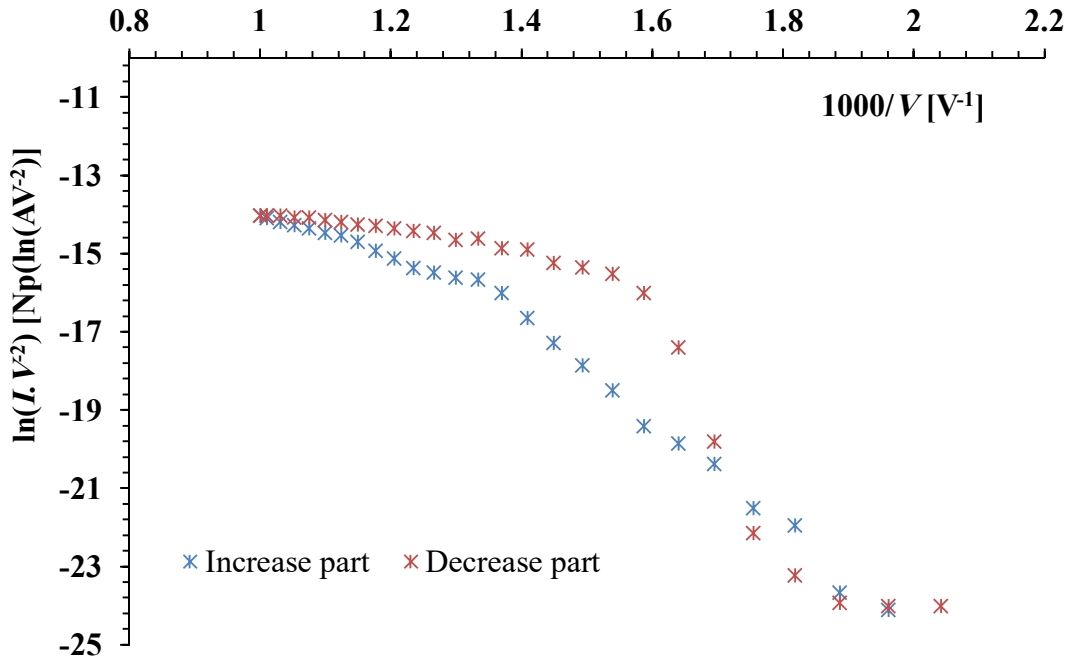

FIG. 5. The Fowler-Nordheim plots for the current-voltage characteristics presented in Fig. 4.

TABLE 1. The results of testing and analyzing the uncoated emitter.

Voltage range [V]	Slope [Np.V]	ζ_C [nm]	γ_C	A_f^{SN} [μm^2]
550 – 610	-10500	166.71	59983.06	32.22

The emitted electrons are incident on a fluorescent screen that lies about 10 mm from the tip. The incident electrons result in light spots on the screen, which can be photographed and compared for characterization purposes. Fig. 6 shows those images for the uncoated tip when using different applied voltages.

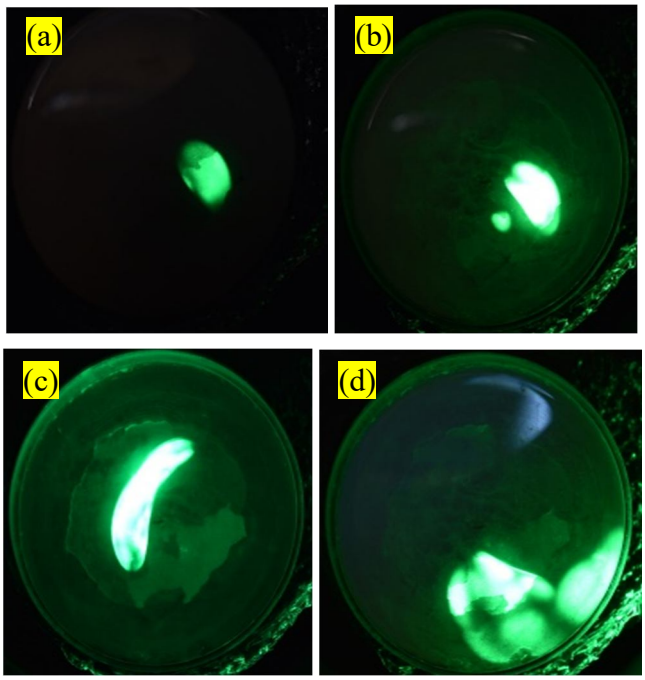

FIG. 6. Images of field-emission currents for the uncoated tungsten tip. The $I_m(V_m)$ data for each photograph is: (a) (630 V, 1.5 nA), (b) (710 V, 30 nA), (c) (850 V, 240 nA) and (d) (990 V, 750 nA).

3.2 Characteristics of Composite Tungsten-Polyethylene Tips

The clean tungsten tip was coated with a 26.9-nm polyethylene layer. The applied voltage was slowly increased across the emitter until the "switch-on" phenomenon is observed. At this voltage (V_{sw}), the emission current switches on from a zero-value to a stable saturated value (I_{sat}) which is known as the switch-on current. In this study, the switch on voltage was 2100 V and the saturation current was 3.5 μA. Fig. 7(a) shows the $I_m(V_m)$ characteristics for the first voltage-decreasing interval. It shows the switch-on current-voltage data, with a voltage range (2100 – 250 V) and a current range (3.5 μA – 80 pA). Fig. 7(b) presents the related Fowler-Nordheim plot, which is a little noisy with a slope value of –0.3811 Np.kV when using a voltage range (450 – 800) Volts.

Figs. 8(a) and 8(b) show the $I_m(V_m)$ characteristics for another cycle obtained from the coated sample for both the voltage increase and decrease intervals, respectively. In the voltage increase cycle, the voltage ranges from (250 – 980 V), where the emission currents range from 10 pA to 2.24 μA. For the voltage decrease cycle, the applied voltages range from (980 – 250 V) with emission currents ranging from 2.3 μA to 90 pA. Fig. 9 shows the related FN plots for the full cycle, noting that the analysis and the orthodoxy test will be carried out later in future research after analyzing the PE layer to know its effective local work-function value.

The stabilities of the field-emission currents of the composite sample are depicted in Figs. 10 (a-d). These figures are obtained during the first decrease interval, where the applied voltages range from (980 – 700 V). To stabilize the emission current, a waiting period of about 10 minutes was adopted before taking each of these photos.

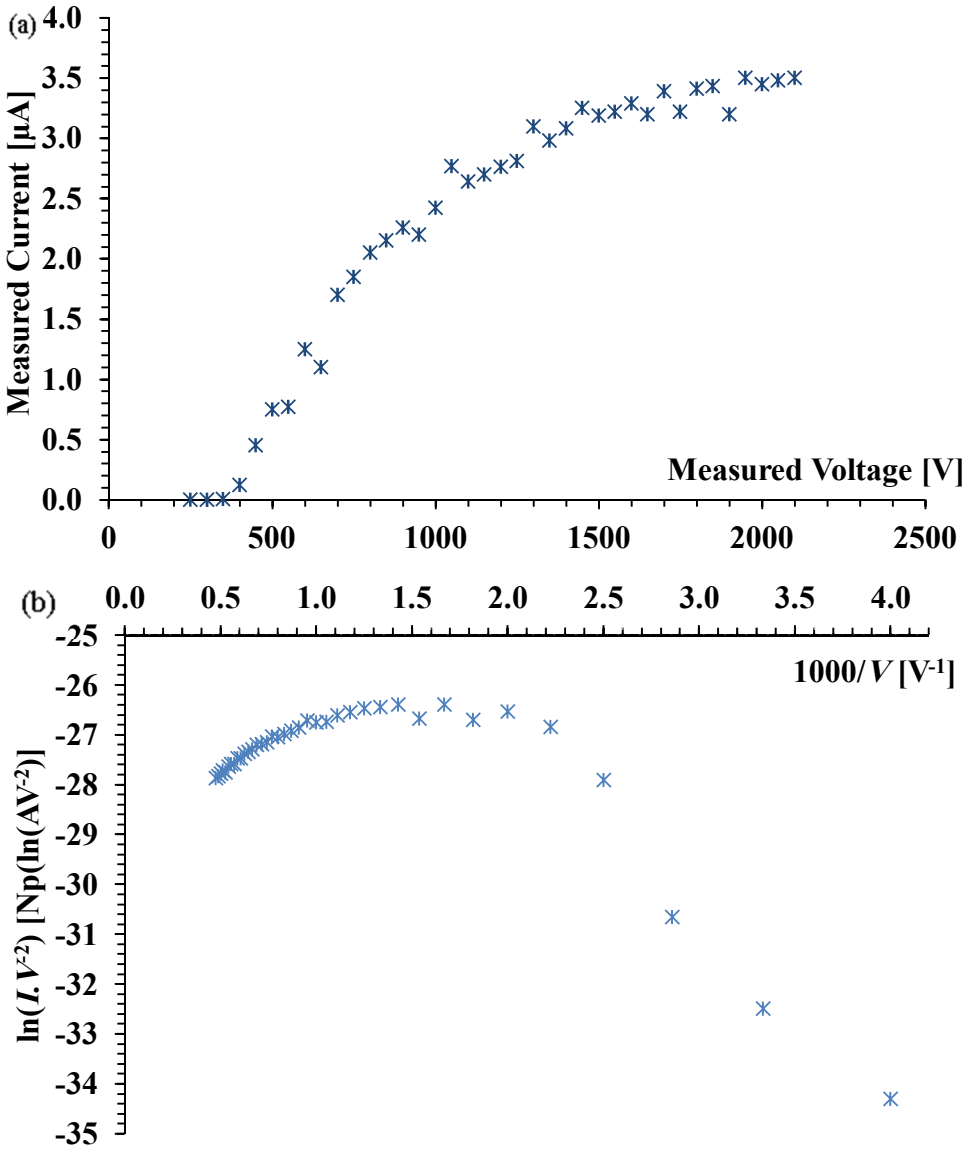

FIG. 7. (a) The current-voltage characteristics for the composite sample during the first voltage-decreasing interval with V_{sw} =2100 V, I_{sat} =3.5 μA and (b) The related Fowler-Nordheim plot.

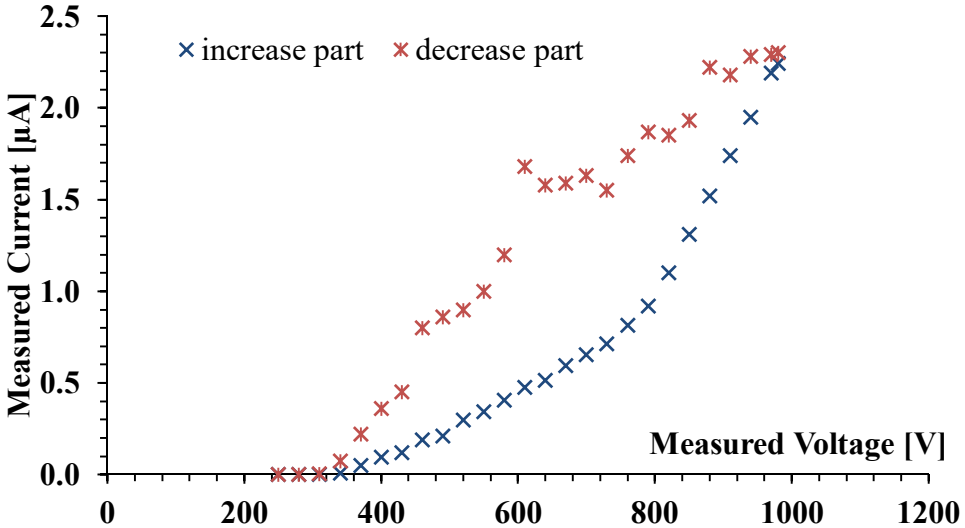

FIG. 8. The current-voltage characteristics for a full testing cycle obtained from a composite sample. (a) Voltage-increasing interval and (b) Voltage-decreasing interval.

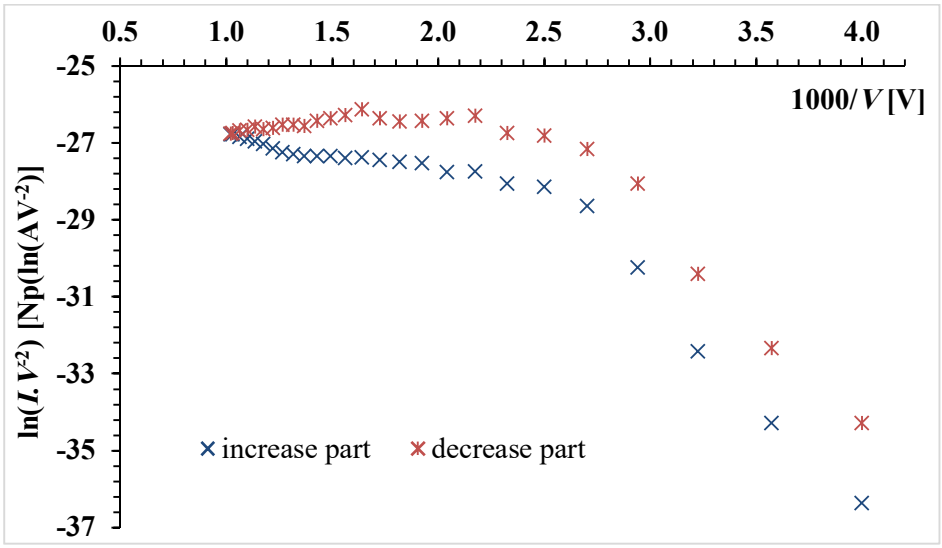

FIG. 9. The Fowler-Nordheim plots for the current-voltage characteristics presented in Fig. 8.

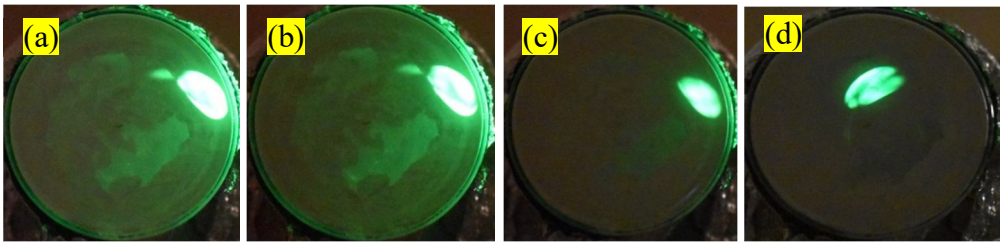

FIG. 10. Field-emission microscope images obtained for the composite sample during the first decrease interval. The current-voltage data for each image is: (a) (980 V, 2.3 μA), (b) (880 V, 2.22 μA), (c) (790 V, 1.87 μA) and (d) (700 V, 1.63 μA).

4. Discussion

Similar $I_m(V_m)$ measurements were obtained for another set of clean and composite samples, as illustrated in Table 2 and Table 3. The results show noticeable improvements in the performance of the emitter after being coated with polyethylene, as shown in the FEM current distribution images (Fig. 6 and Fig. 10). The differences in the performance among the samples are related to two main factors; namely, the apex radius of the clean sample and the thickness of the polyethylene layer. Also, the performance of the clean field electron emitter could be affected by the contamination layers that may occur while transporting and installing the samples to the field electron microscope or during the pumping process. Effects of contamination on the electronic-emission process were extensively reviewed by Latham *et al*. [1].

TABLE 2. Characteristics of clean (uncoated) samples.

Sample Number	Applied-voltage range (V)	Emission-current range (nA)	The radius of the tip (nm)
1	450 – 940	0.0065 – 780	76.3
2	490 – 1000	0.001 – 820	85.8
3	720 – 1280	0.006 – 680	90.5
4	1020 – 2070	0.007 – 615	101.1

TABLE 3. Characteristics of composite (coated) samples.

Sample Number	Switch-on voltage (V)	Switch-on current (μA)	Applied-voltage range (V)	Emission-current range (nA)	The radius of the tip (nm)	Thickness of polyethylene layer (nm)
1	1800	4.8	290 – 850	0.095– 4100	106.5	30.2
2	2100	3.5	250 – 980	0.01– 2240	112.7	26.9
3	2400	2.3	400 – 900	0.08 – 1900	119.8	29.3
4	3500	1.4	500 – 1300	0.075 - 1200	128.7	27.6

The appearance of concentrated single light spot in the field-electron microscope image is a signature of the uniform distribution of the polyethylene layer on the emitter apex and is thought to be associated with the formation of a "conducting channel" through the polyethylene layer, as illustrated schematically in Fig. 11[1]. The physics of this behavior can be explained in the context of forming a conductive semi-crystallized channel between the tip apex and polyethylene /vacuum surface. This channel will act as a conductive medium, but will protect the core emitting tip from ion back bombardment which normally damages the emitting tip after some time.

On the contrary, the appearance of scattered multi-spot FEM images is associated with the inhomogeneity in the distribution of the polyethylene-layer and/or with substrate irregularities that may lead to the formation of multiple emitting channels [1, 24, 25].

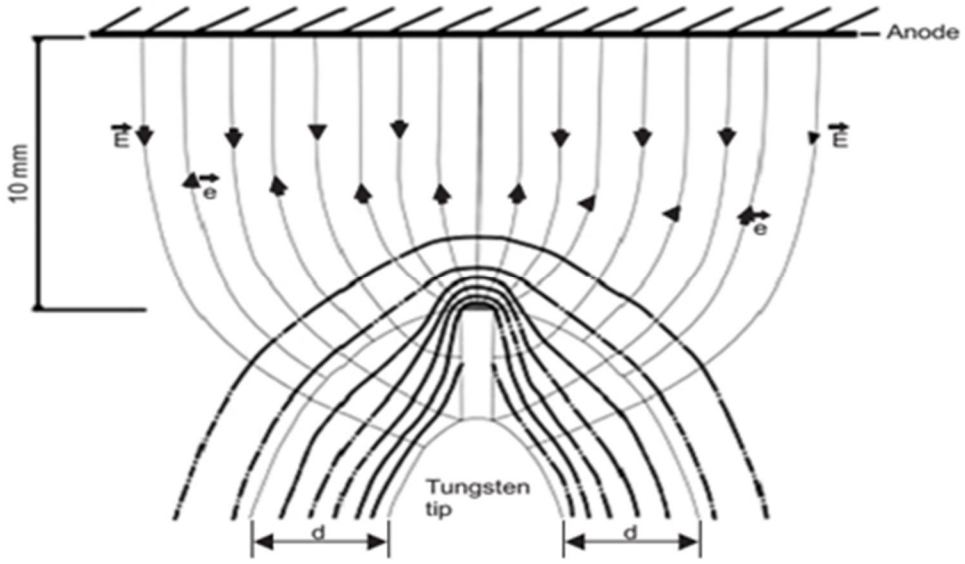

FIG. 11. Schematic representation of an emitting channel formed in the dielectric layer [1].

5. Conclusions

This experimental work addresses the effects of polyethylene coating on the field-electron emission characteristics of clean tungsten tips. Several tungsten tips, with different apex radii, have been prepared, coated with different thicknesses of polyethylene layers and then examined in a standard field-electron microscope. Various field-electron emission characteristics have been measured under high vacuum conditions, before and after coating the tips with polyethylene. These include current-voltage characteristics, Fowler-Nordheim analysis plots, scanning electron micrographs and spatial current distributions (electron emission images). Based on this work, it is proved that coating tungsten tips with polyethylene layers causes noticeable improvements on the tip emission properties. In particular, coating the tips improves the current-voltage characteristics, which is reflected in lowering the extraction voltage, getting more stable emission currents and finally, the spatial distributions of the emitted electrons for the coated tips are more stable and uniform.

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