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ARTICLE

Composite Metallic Nano Emitters Coated with a Layer of Insulator Covered by Au Layer

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Abstract: In this work, the differences in the behavior and properties of the emitted electron beam from tungsten (W) tips were studied before and after coating these tips with a thin layer of dielectric material followed by a thin layer of gold, to improve the emission current density, stability and emission current pattern concentration. The core of the composite cathode is made of high-purity tungsten (W). Measurements have been made with clean W emitters before and after coating these tips with two types of epoxy resins (epoxy 478 resins or epoxy UPR- 4 resins) followed by a thin layer of gold. For critical comparison, several tungsten tips with various apex-radii have been prepared using electrochemical etching techniques. The emitters have been coated by dielectric thin films of various thicknesses and the layer of Au used for coating the Epoxy layer has the same thicknesses. Their behavior has been recorded before and after the process of coating. These measurements include the current-voltage (I-V) characteristics and Fowler-Nordheim (F-N) plots. Imaging has also been done using a visible light microscope (VLM), along with a scanning electron microscope (SEM) to help in characterizing the epoxy layer thickness on the tip surface after coating. Besides, the emission patterns have been recorded from the phosphorescent screen of a field electron emission microscope (FEM). Having two types of composite systems tested under similar conditions provided several advantages. These measurements helped in producing a new type of emitters that have more suitable features with each of the two resins.

Keywords: Cold field emission, Nano emitter, Dielectric coating, Au layer.

Introduction

The improved electron emission from metal through dielectric and metallic coatings has been a subject of considerable interest at present. Within the search for stable and bright electron sources, a deeper understanding of high voltage breakdown phenomena has been greatly required by developing theories of induced electron emission in the metal-insulator-metal-vacuum interface. The emitter is usually formed into a tip, which has an apex radius ranging from several nanometers to a hundred nanometers [1-4]. When preparing very sharp emitters, it is necessary to use a metal of the highest possible quality. Within this work, all experiments have been carried out using high-purity polycrystalline tungsten wire. Tungsten is used due to its favorable properties, such as the highest melting point, high strength, high work function and heat resistance at high temperatures [1, 3- 7]. Moreover, tungsten emitters with different apex radii ranging from 100 -150 nm have been prepared and coated with a thin layer

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of various epoxy resin types (478 and UPR-4 resins) [1, 4- 7]. Additionally, the Epoxy layer has been covered by a thin layer of gold. The thickness of the epoxy layer was approx. 150 nm. The scanning electron microscope was used to extract the thickness of the epoxy layer. The thickness of the Au layer was 4 nm determined by the sputter coater. The emission patterns have been directly recorded from the phosphor screen of the field electron microscope (FEM) using a digital camera. The current-voltage (I-V) measurements and Fowler–Nordheim (F-N) plots have been obtained under ultra-high vacuum (UHV) conditions with a basic pressure of about 10^{-7} Pa.

Experimental Techniques

The tungsten emitters (tips) were prepared by electrolytic etching. A 0.1 mm diameter tungsten wire was immersed into a 2 molar sodium hydroxide solution with the emitter held by a conductive cylinder usually made of corrosion-proof material. Both the anode (tungsten wire) and the cathode (steel cylinder) were immersed in the sodium hydroxide solution and connected to a DC power source (12 V). When immersing approximately 0.8 cm of tungsten wire in NaOH solution, the etching current starts with a value of around (5 μA). Then, the tip is placed in an ultrasonic bath for 15 minutes to clean it from the oxide layers formed on the tip surface [1, 6-10].

We have discovered that to obtain a layer of 150 nm thick epoxy layer (Epoxylite 478 resin or Epoxylite UPR-4 resin) on the emitter surface, the tip has to be dipped into the epoxy 12 times very slowly and vertically, which yields a 15 nm layer [1, 8, 11]. In the following way, the sample holder that keeps the sample in a vertical position is mounted on a trolley that moves vertically and lowers to a flask of Epoxylite resin and the tip [7, 10]. To ensure an even distribution of resin on the surface of the tip and stabilize the Epoxylite resin on the tip surface, the coated tip is transferred into a furnace and subjected to 30-minute curing at 373 K to drive off the solvent, followed by 30-minute curing at 453 K to complete the curing of the resin [4, 7-10]. Then, we used the sputter coater to cover the tips by 4nm of Au, where a desired operating pressure has been obtained by a rotary pump with an inert gas, such as argon, admitted to the chamber by a fine control valve to ionize the argon gas. It also applies an electric field which accelerates the ions to the tip, as shown in Fig. 1 [9-14].

Next, the composite emitter is mounted in a standard FEM with an emitter screen distance of 10 mm [1, 4, 10- 13]. The emission patterns are recorded from a phosphor screen coated by tin oxide layers. All the experiment was performed under ultra-high vacuum (UHV) system pressures of approx. 10^{-7} Pa. The UHV system has been baked at a temperature of about 453 K for 12 hours [1, 13- 18]. Before adding the liquid nitrogen to the trap, the radius of each emitter apex has been measured from an image taken in a 30 kV SEM at magnifications up to 1000 X.

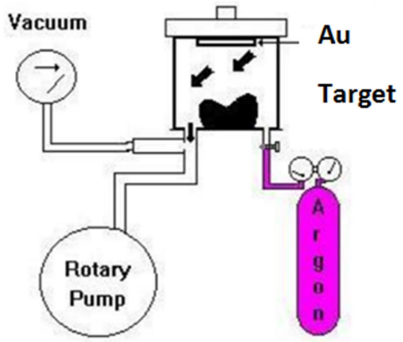


FIG.1. A schematic diagram showing the sputter coater.

Results and Discussion

The tungsten micro emitters have been prepared to have an apex radius of approximately 100 to 150 nm. Presented results include SEM images [14, 15] of emitter's apex, as well as I-V characteristics and F-N type plots of the field emission characteristics.

Fig. 2A, B shows the visible light microscope (VLM) images for uncoated type 1 and type 2 emitters. The measurement radii for emitter types 1 and 2 were 125 nm and 120 nm, respectively. Fig. 3A shows SEM images for emitter type 1 which had an approximately hemispherical apex being coated with a 150 nm thick layer of the Epoxylite UPR- 4 resin followed by 4 nm of gold. Fig. 3B shows a SEM image for emitter type 2 which had an approximately hemispherical apex being coated with a 150 nm thick layer of Epoxylite 478 resin followed by 4 nm of gold.

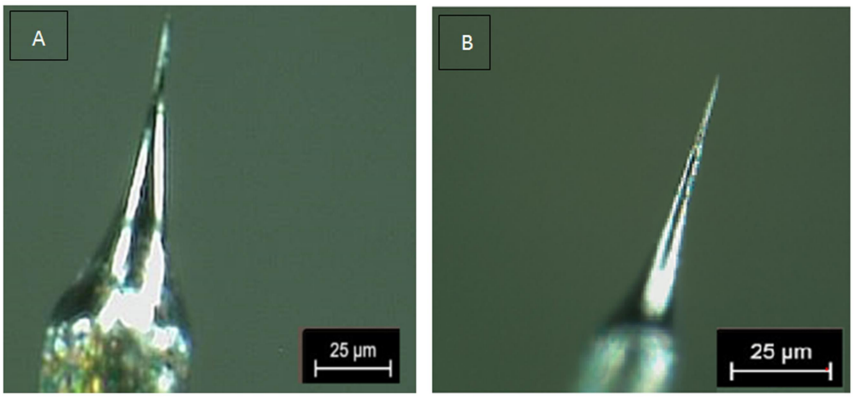


FIG. 2. A) Visible light microscope image (X 1500) for uncoated emitter type 1, with an apex radius of 125 nm. B) Visible light microscope image (X 1500) for uncoated emitter type 2, with an apex radius of 120 nm.

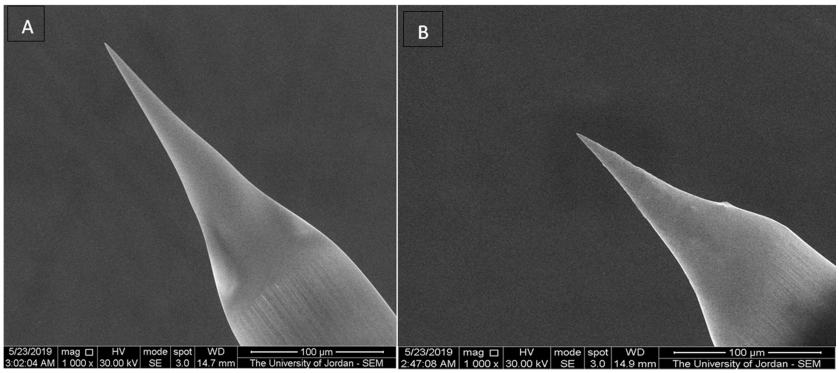


FIG. 3. A) SEM image at magnification (X 1000) for emitter type 1(tungsten/ 150 nm UPR- 4 resin/ 4 nm Au). B) SEM image at magnification (X 1000) for emitter type 2 ((tungsten/ 150 nm 478 resin/ 4 nm Au).

The most important factors to be considered are the structure and stability of the emission pattern, as these effects are a reflection of the influence of the intermediate coating on the emission properties. The emission of two types of newly synthesized emitters was tested and photographed on the phosphorus emission screen. The emission current patterns for the uncoated emitters have been taken at the same voltage values when the voltage was decreased slowly. In detail, the FEM emission current pattern primarily consists of multi bright spots. Fig. 4 shows the emission current pattern for uncoated emitter type 1. Fig. 5 shows the emission current pattern for uncoated emitter type 2. Fig. 6 shows the stability structure pattern for uncoated emitter type 1. Fig. 7 shows the stability structure pattern for uncoated emitter type 2. However, the stability structure images for the two types of emitter were taken at the same voltage and the duration time between obtaining the next images is 15 minutes.

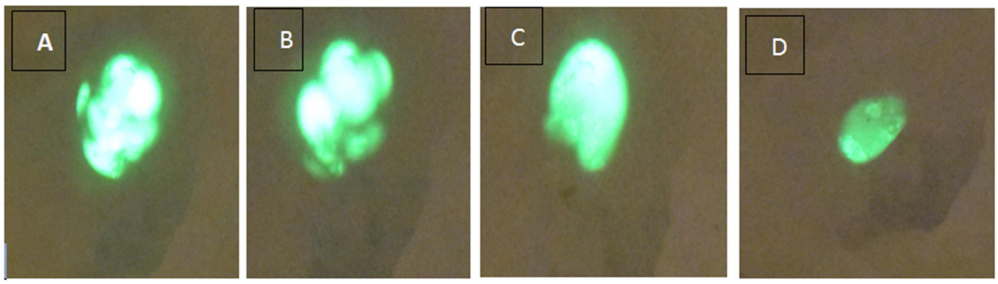


FIG. 4. Emission current pattern for uncoated emitter type 1. A) Emission current 1.5 µA, applied voltage 1600 V. B) Emission current 1.2 µA, applied voltage 1400 V. C) Emission current 1 µA, applied voltage 1200 V.D) Emission current 0.4 µA, applied voltage 1000 V.

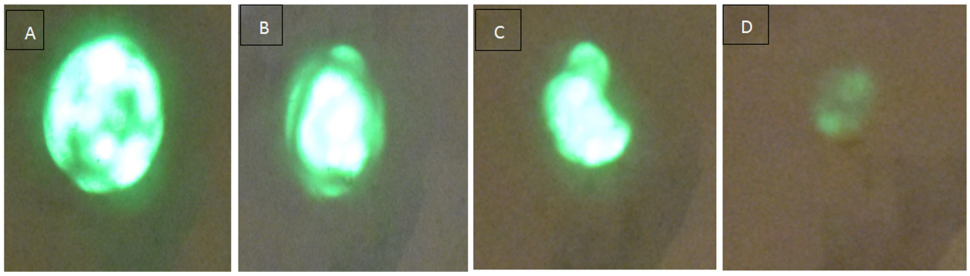


FIG. 5. Emission current pattern for uncoated emitter type 2. A) Emission current 1.8 μA, applied voltage 1600 V. B) Emission current 1.6 μA, applied voltage 1400 V. C) Emission current 1.3 μA, applied voltage 1200 V. D) Emission current 0.8 μA, applied voltage 1200 V.

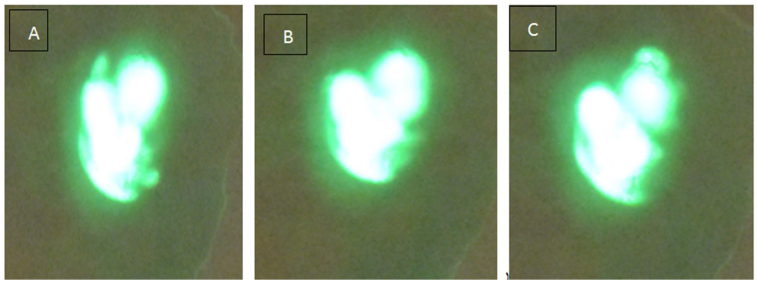


FIG. 6. The stability structure pattern for uncoated emitter type 1, at emission current 2.1 μA and applied voltage 1800 V; the duration time between obtaining the next images is 15 minutes.

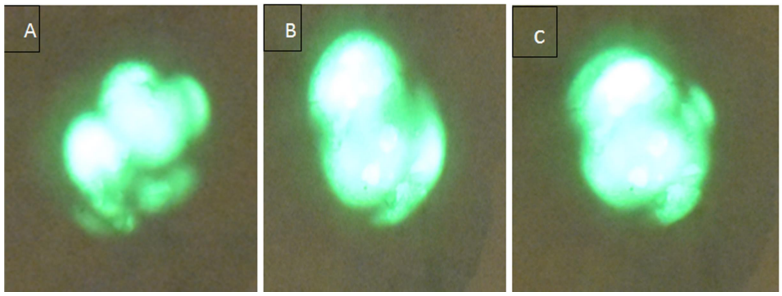


FIG. 7. The stability structure pattern for uncoated emitter type 2, at emission current 2.3 μA and applied voltage 1800 V; the duration time between obtaining the next images is 15 minutes.

The emission current patterns for coated emitters were taken at the same voltage values. The images were taken for the emission current patterns at the switch-on voltage and then, the voltage was regularly decreased, where the FEM image primarily consists of a single bright spot. Fig. 8 shows the emission current pattern of emitter type 1 after being coated with a 150 nm thick layer of Epoxylite UPR- 4 resin followed by 4 nm of Au. Fig. 9 shows the emission current pattern of emitter type 2 after being coated with a 150 nm thick layer of Epoxylite 478 resin followed by 4 nm of Au. The stability structure pattern for emitter type 1 after being coated with Epoxylite UPR- 4 resin followed by 4 nm of Au is shown in Fig. 10. The stability structure pattern for emitter type 2 after being coated with Epoxylite UPR- 4 resin followed by 4 nm of Au is also shown in Fig. 11. However, the stability structure patterns for the two types of emitters have been taken at the same voltage and the duration time between obtaining the next images is 15 minutes.

From the comparison of the emission patterns, emission pattern stability and the SEM image show the tip geometry and surface. With that, it has been concluded that the emission current pattern and stability structure for the emission current from emitter type 2 (tungsten/ 150 nm 478 resin/ 4 nm Au) is more stable in comparison with the emission current from emitter type 1 (tungsten/ 150 nm UPR-4 resin/ 4 nm Au). The emitted electrons were concentrated in one single spot with high brightness, where a single bright spot formed. Also, the electrons will be emitted from the epoxy layer in different proportions, depending on the components of each type of epoxy.

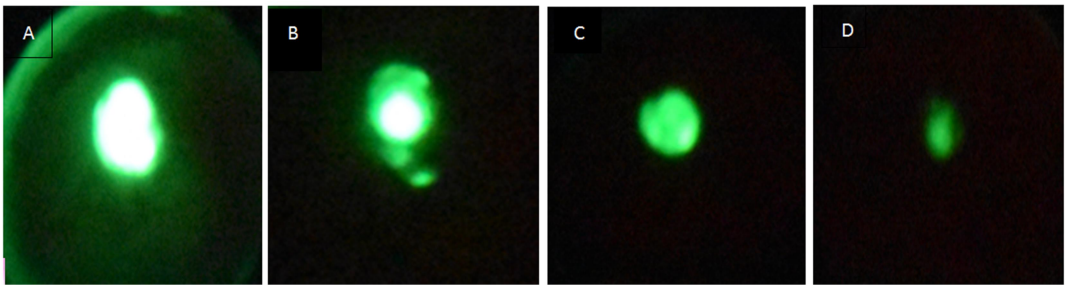


FIG. 8. Emission current pattern for emitter type 1 (Tungsten/ 150 nm UPR-4 resin/ 4 nm Au).A) Emission current 7 μA, applied voltage 1600 V. B) Emission current 4 μ A, applied voltage 1400 V. C) Emission current 3.6 μ A, applied voltage 1200 V. D) Emission current 2.4 μ A, applied voltage 1000 V.

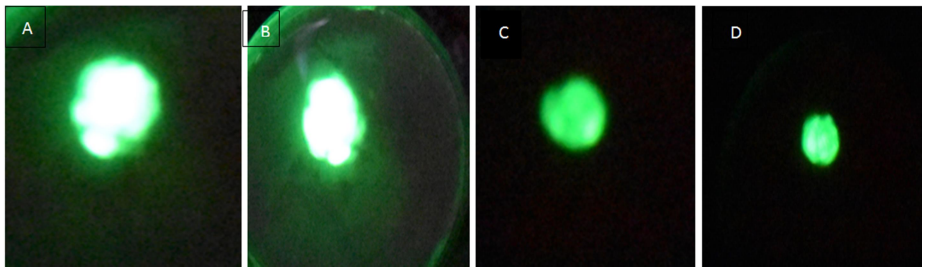


FIG. 9. Emission current pattern for emitter type 2 (Tungsten/ 150 nm 478 resin/ 4 nm Au) A) Emission current 10.6 μ A, applied voltage 1600 V. B) Emission current 9.4 μ A, applied voltage 1400 V. C) Emission current 8.3 μ A, applied voltage 1200 V. D) Emission current 2.5 μ A, applied voltage 900 V.

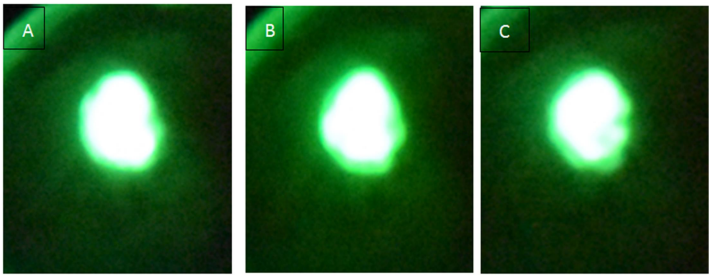


FIG. 10. The stability structure pattern for emitter type 1 (Tungsten/ 150 nm UPR-4 resin/ 4 nm Au), at emission current 10 μA and applied voltage 1800 V. The duration time between obtaining the next images is 15 minutes.

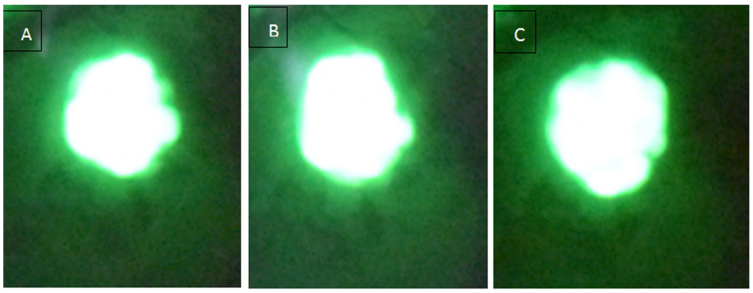


FIG. 11. The stability structure pattern for emitter type 2 (Tungsten/ 150 nm 478 resin/ 4 nm Au), at emission current 13 μA and applied voltage 1800V. The duration time between obtaining the next images is 15 minutes.

The (I-V) plot shows the total emission of current on voltage V externally applied between cathode and anode. Fig. 12A shows the I-V characteristics for uncoated emitter type 1. This typical curve for the non-ohmic behavior for tungsten emitter has been cleaned by alcohol, distilled water and ultrasonic bath. Fig. 12B shows the I-V characteristics for emitter type 1 after being coated with 150 nm Epoxylite 478 resin. There were obvious fluctuations of emission current while the voltage applied has been slowly applied throughout the emitter. At a certain threshold switching voltage value, Vsw = 1600 V, the emission current starts from zero to 2 μA, by decreasing the voltage of the line region of F-N plot that extends down to V_{SAT} = 1300 V with I_{SAT} = 1.1 μA, by decreasing the voltage of the emission current that vanishes at V_{TH} = 600 V with emission current I_{Th} = 56.3 PA. Fig. 12C shows the I-V characteristics after

being coated with 150 nm with Epoxylite UPR-4 resin followed by 4 nm of Au, where a switch-on phenomenon has been observed without detecting current fluctuations before switching the threshold switching voltage V_{SW} = 1400 V and switching current I_{SW} = 5 µA, by decreasing the voltage of the line region of F-N plot that extends down to V_{SAT} = 700 V with I_{SAT} = 1.17 µA, by decreasing the voltage of the emission current that vanishes at V_{TH} = 500 V with emission current I_{Th} = 86.4 PA.

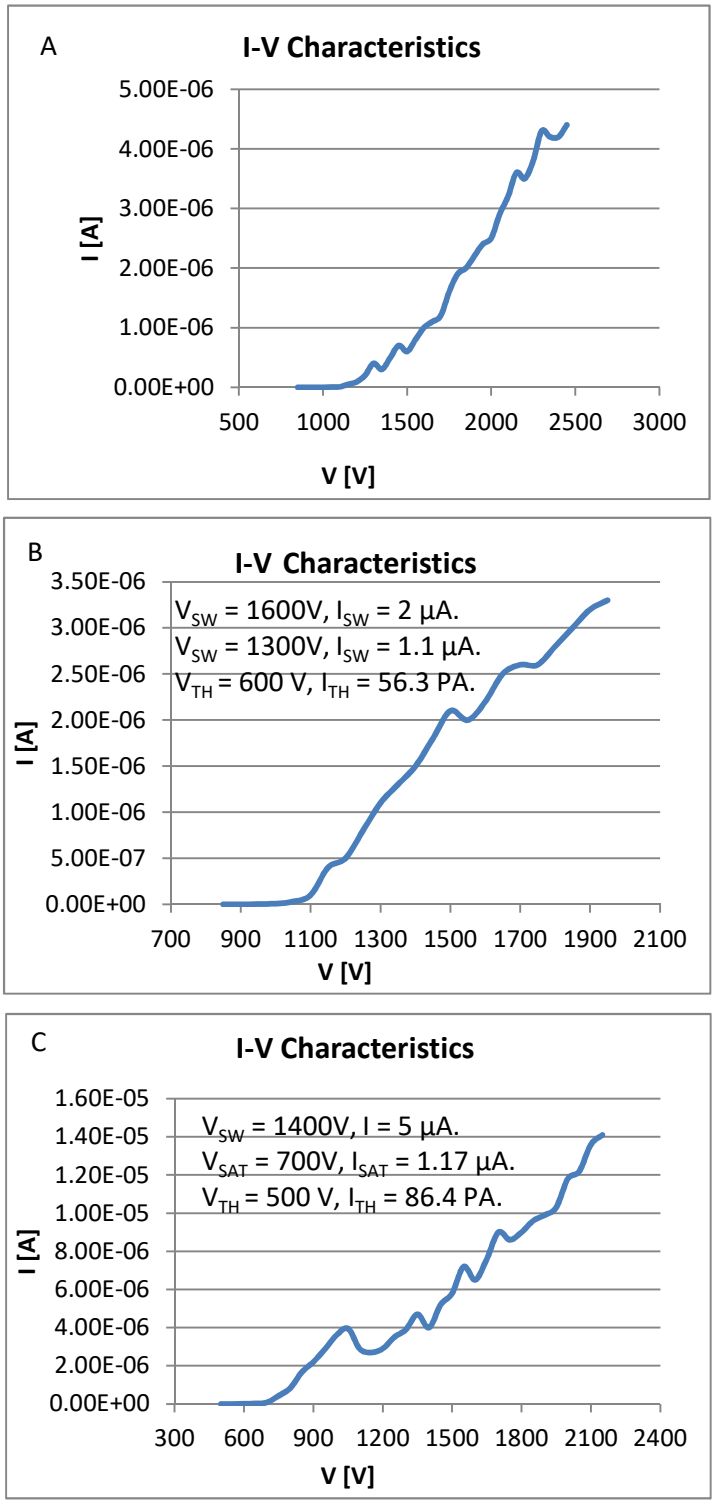
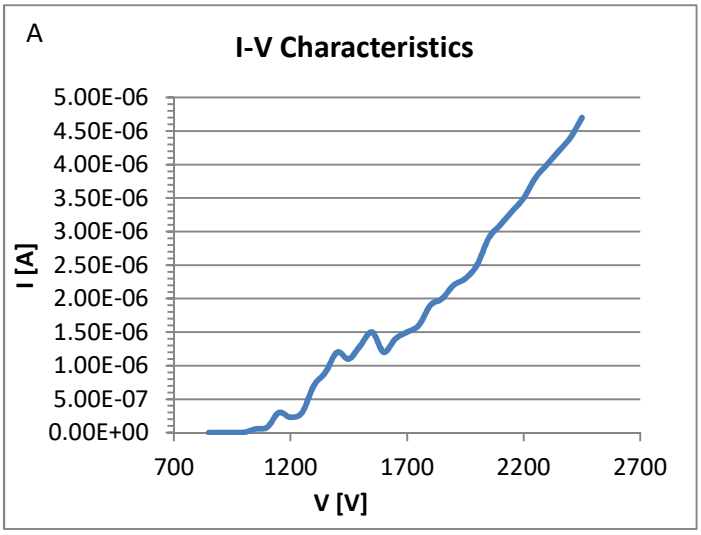
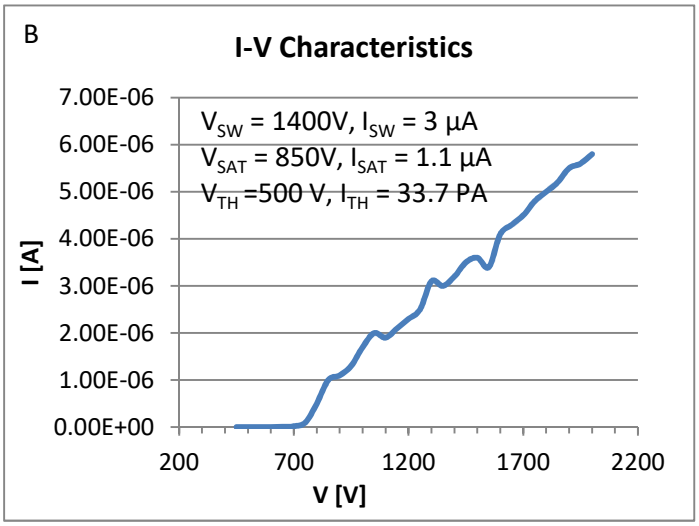


FIG. 12. The I-V characteristics. A) Uncoated emitter type 1. B) Emitter type 1(Tungsten/ 150 nm 478 resin), V_{SW} = 1600 V with I_{SW} = 1.7 µA, V_{SAT} = 1300 V with I_{SAT} = 1.1 µA, V_{TH} = 600 V, I_{TH} = 56.3 PA. C) Emitter type 1 (Tungsten/ 150 nm UPR- 4 resin/ 4 nm Au), V_{SW} = 1400 V with I_{SW} = 5 µA, V_{SAT} = 850 V with I_{SAT} = 1.67 µ A.

Fig. 13A shows the I-V characteristics for uncoated emitter type 2, as this is a typical curve for the non-ohmic behavior. Tungsten emitter has been cleaned by alcohol, distilled water and ultrasonic process. Fig. 13B shows the I-V characteristics for emitter type 2 after coating with 150 nm Epoxylite 478 resin. There were obvious fluctuations of emission current, while the voltage has been slowly applied throughout the emitter. At a certain threshold switching voltage value, Vsw = 1400 V the emission current starts from zero to 3 µA, by decreasing the voltage of the line region of F-N plot that extends down to V_{SAT} = 850V with I_{SAT} = 1.1 µA, by decreasing the voltage of the emission current that vanishes at V_{TH} = 500V with I_{TH} = 33.7 PA Fig. 13C shows the I-V characteristics after coating with 150 nm Epoxylite 478 followed by 4 nm of gold, where a switch-on phenomenon has been observed without detecting current fluctuations before switching the threshold switching voltage V_{SW} = 1300 V and the switching current I_{SW} = 9µA, by decreasing the voltage of the line region of F-N plot that extends down to V_{SAT} = 650V with I_{SAT} = 1.12 µA, by decreasing the voltage of the emission current that vanishes at V_{TH} = 350V with I_{TH} = 65.9 PA.





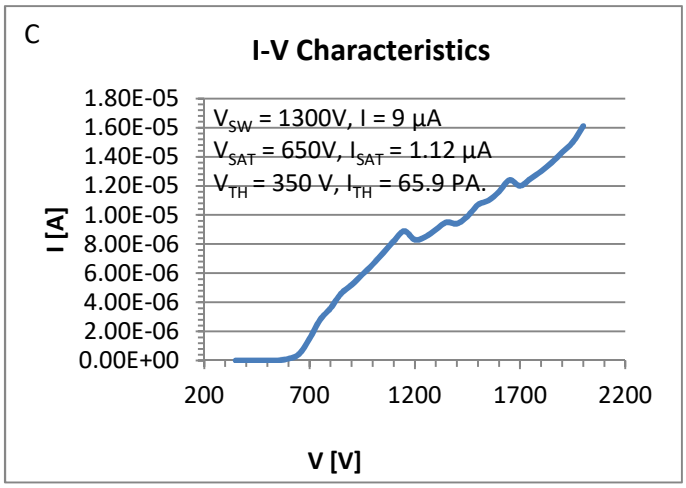


FIG.13. The I-V characteristics for A) Uncoated emitter type 2. B) Emitter type 2 (Tungsten/ 150 nm 478 resin), V_{SW} = 1400 V with I_{SW} = 3 µA, V_{SAT} = 850 with I_{SAT} = 1.1 µA, V_{TH} = 350 V, I_{TH} = 65.9 PA. C) Emitter type 2 (Tungsten/ 150 nm 478 resin/ 4 nm Au), V_{SW} = 1300 V with I_{SW} = 9 µA, V_{SAT} = 700 V with I_{SAT} = 1.12 µA, V_{TH} = 350 V, I_{TH} = 65.9 PA.

The study of the second approach of I-V data is conducted through the Fowler-Nordheim (FN) plot *via* $\ln(\frac{I}{V^2})$ *versus* $(\frac{1}{V})$, as shown in Fig. 14. Curve (A) shows the characteristics of clean tungsten emitter type 1, while curve (B) shows the characteristics of tungsten emitter after being coated with 150 nm Epoxylite UPR- 4 resin. However, curve (C) shows the characteristics of tungsten emitter after being coated with 150 nm Epoxylite UPR- 4 resin followed by 4 nm Au. Curve (A) of Fig. 15 shows the characteristics of clean tungsten emitter type 2, while curve (B) shows the characteristics of tungsten emitter after being coated with a 150 nm thick layer of Epoxylite 478 resin. Curve (C) shows the characteristics of tungsten emitter after being coated with a 150 nm thick layer of Epoxylite 478 resin followed by 4 nm Au.

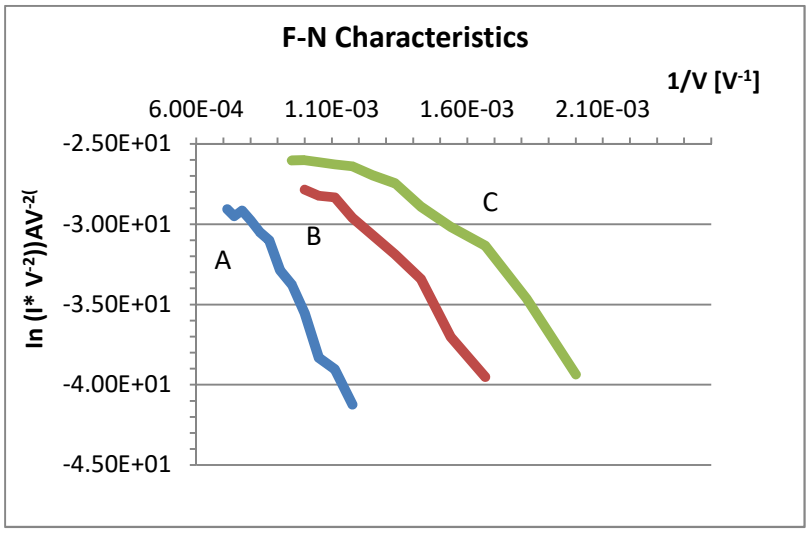


FIG. 14. The F-N plot for emitter type 1, where curve A is for clean emitter, curve B after being coated with a 150 nm thick layer of Epoxylite UPR- 4 resin and curve C after being coated with a 150 nm thick layer of Epoxylite UPR- 4 resin followed by 4 nm of Au.

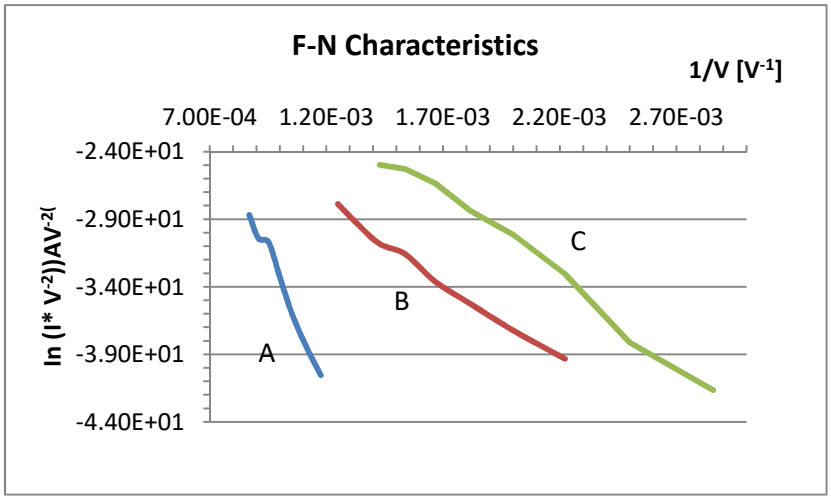


FIG. 15. The F-N plot for emitter type 2, where curve A is for clean emitter, curve B after being coated with a 150 nm thick layer of Epoxylite 478 resin curve C after being coated with a 150 nm thick layer of Epoxylite 478 resin followed by 4nm of Au.

The new composite micro emitters covered by Epoxylite resin and followed by Au layer had the following effects: A) The switch-on voltage for the emitter after being coated with epoxy layer followed by Au layer is lower than the switch-on voltage for clean emitters and the emitter is coated with epoxy layer only with high emission current. B) The F-N plots are approximately linear for emission current below 10^{-7} A with a duration of higher currents. C) The emission patterns are in the form of a very regular and concentrated spot more than the emission patterns for the clean emitter and the emission patterns for coated emitter with the epoxy layer only.

Conclusion

Details of new types of composite microstructure have been presented. The results confirmed that the excellent field emission behaviour of the coated emitter tip is a prospective candidate for advanced electron field emitters. The most important finding from this work is that the switch-on phenomenon occurs with the 2 types of the epoxy resin that were used even covered with the Au layer. Another important finding is that the emission patterns, at the gross level, are in all cases relatively stable, as this is a necessary requirement if an emitter is to be used as an electron source. The switch-on voltage and the threshold switch-voltage have been decreased after coating the emitters with a dielectric material even after being covered with a thin layer of Au. This has been accompanied by a rise in the value of the emission current and stability in the electrons' emission patterns, where the electrons have been centered in the form of a bright spot. The field emission characteristics of a tungsten electron source are intrinsically changed by coating the tips with a micro-thin layer of epoxy resin covered with a thin layer of Au. This is in line with the results obtained from similar studies [19]. This change in the characteristics varied depending on the type of epoxy resin used in coating the tungsten tips.

Acknowledgments

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