

Characterization of Composite Electron Sources (Metal - Insulator - Vacuum)

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Abstract: Field electron emission measurements were performed on composite tips (Insulator- Tungsten) that were prepared by electrochemical etching in NaOH solution. The current-voltage (I-V) characteristics and field electron emission images were recorded under UHV conditions with a base pressure of $<10^{-9}$ mbar. Next, the tips were coated with several different types of dielectric layers. Various techniques were employed to measure the characteristics of these types of emitters and to evaluate the effects of different types of dielectric coatings on the performance and reliability of such an electron source.

Keywords: Field electron emitter, Dielectric layers, Composite tip, Field electron microscope.

Introduction

Electron sources employing field emitters have not been reported until 1954 [1, 2]. The first utilization of the field electron emission mechanism appeared lately in 1968, as part of a scanning electron microscope [1, 2]. Field electron emission (FEE) is the emission of electrons from the surface of a cathode under the influence of a high applied electrostatic field (typically about 3 V/nm) [3, 4]. Considerable experimental studies of field emission have been carried out on carbon fibers, tungsten (W) and lanthanum hexaboride [5]. The most widely used material for field emitters is tungsten [6, 7]. Tungsten brings several benefits due to its favorable properties, such as the high melting point of 3377 °C, a work function of 4.55eV, high hardness (strength) and appropriate heat

resistance at high temperatures [8]. Techniques for preparing field emission cathodes have been developed. Advances in vacuum technology have also been important, since a high-quality vacuum is essential for reliable operation of field-emission cathodes. These changes have enabled the widespread use of field-emission cathodes [9, 10]. Cathode-manufacturing technology based on electrolytic etching [11] has been studied and enhanced. This technology makes it possible to prepare a cathode tip with a diameter of several nanometers [12]. Composite micropoint cathodes have been prepared to avoid degradation of the electron emitter due to ion sputtering processes during emission for long lifetime and to improve the emission characteristics [5, 13-15, 16]. The research

reported here includes comparing the current-voltage (I-V) characteristics in Fowler-Nordheim (F-N) plots, in addition to determining the spatial emission current distributions and the emission stability (electron emission images) with various dielectrics.

Materials and Methods

The procedures for coating Tungsten (W) tips have been described [12, 15]. In all cases, the microprint nano-apex metallic emitters used for such measurements were electrolytically etched from 0.1mm diameter tungsten wires with 99.95% purity (good fellow metals) using a 2M solution of NaOH. Then, the tip is ultrasonically cleaned. The coating procedure required slowly dipping the tip into the resin and then carefully removing it to ensure that only a thin film remained on the tip surface. Then, the tip is carefully transferred to an oven and subjected to a curing cycle of thirty minutes at 100 °C to drive off the solvents, followed by another thirty minutes at 185 °C to complete curing of the resin [12, 15]. The coated tips were studied in evacuated field emission microscopes with a diffusion pump system having an additional liquid nitrogen trap. Base pressures of 5×10^{-8} mbar have been reached after baking the system at a temperature of 180 °C for 12 hours with more liquid nitrogen added to the trap. The separation between the cathode and the anode was standardized at ~ 10 mm using a current-limiting resistor of 20 M Ω . The images presented were taken by a digital camera through the window of the vacuum system. To record the emission behavior in the vacuum, a high-tension (EHT) power supply is used to apply a negative voltage to the cathode. The emission current is

measured with a Kiethley 485 auto-ranging picoammeter [17].

Results

Tungsten microemitters have been analyzed with several coatings: (1) Clark Electromedical Instruments Epoxylite resin, (2) Epidian 6 (based on Bisphenol A), (3) A Radianox compound of graphite colloid and (4) Molyslip 2001 E compound (MoS₂ and MoS). The results presented include the I-V characteristics, F-N plots and emission images. We compared the results with those obtained with clean uncoated tungsten microemitters.

Composite Tungsten - Clark Electromedical Instruments Epoxylite Resin Tip

First, we determined the emission characteristics of a clean uncoated tungsten tip as shown in I-V characteristics (left) and the Fowler-Nordheim (F-N) plot (right) in Fig. 1. In each case, the potential was slowly increased until the "switch-on voltage" V_{sw} was reached. At this point, the emission current switches on to a stable saturated value which is called the "switch-on current" I_{sat} . Here, $V_{sw} = 2400$ V and $I_{sat} = 1.5$ μ A. Fig. 2 (left) shows the I-V characteristics when decreasing the voltage and switch-on voltage with voltage range (2400 - 700) V and current range (1.5 μ A - 8.8 pA), with the F-N plot on the right side of the same figure. Fig. 3 (left) shows the voltage increasing range (700 - 1900) V and (6.7 pA - 1.1 μ A), with the F-N plot on the right side of the same figure. Fig. 4 shows a comparison between the field emission of the tungsten tip before and after coating by Clark Electromedical Instruments Epoxylite resin.

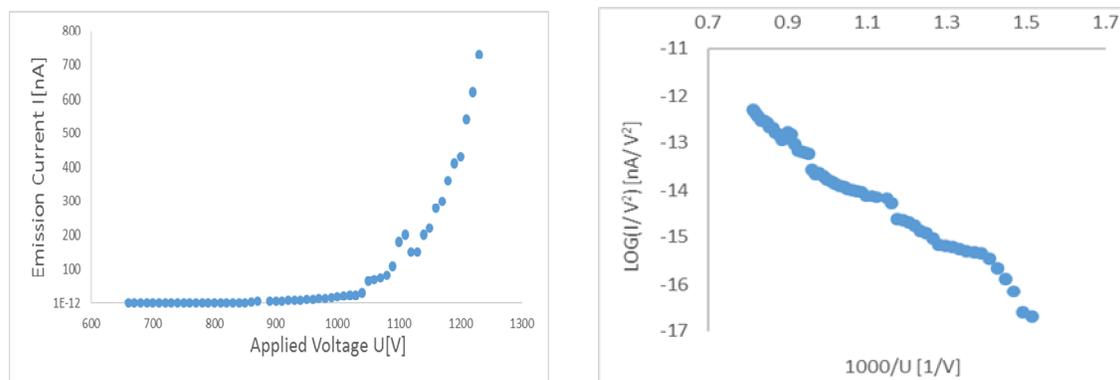


FIG. 1. The I-V characteristics (left) of clean tungsten tip before coating by Clark Electromedical Instruments Epoxylite resin whilst increasing the voltage; and the Fowler-Nordheim plot (right) with a slope of 5500 decades per inverse volt. The applied potential is in the range of (660 - 1230) V.

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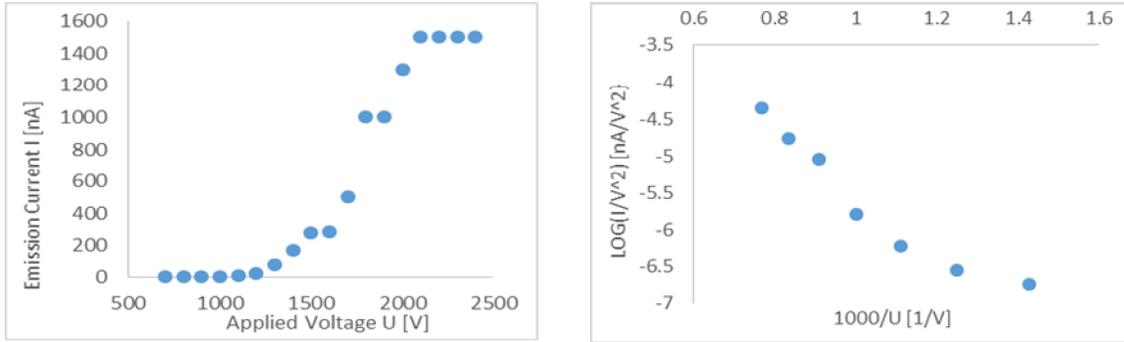


FIG. 2. The I-V characteristics (left) of composite Tungsten - Clark Electromedical Instruments Epoxylite resin during the first decreasing voltage with V_{sw} (2400 V, 1.5 μ A); and Fowler-Nordheim plot (right). The F-N plot slope is 3800 decades per inverse volt. The applied voltage is in the range of (2400 – 700) V.

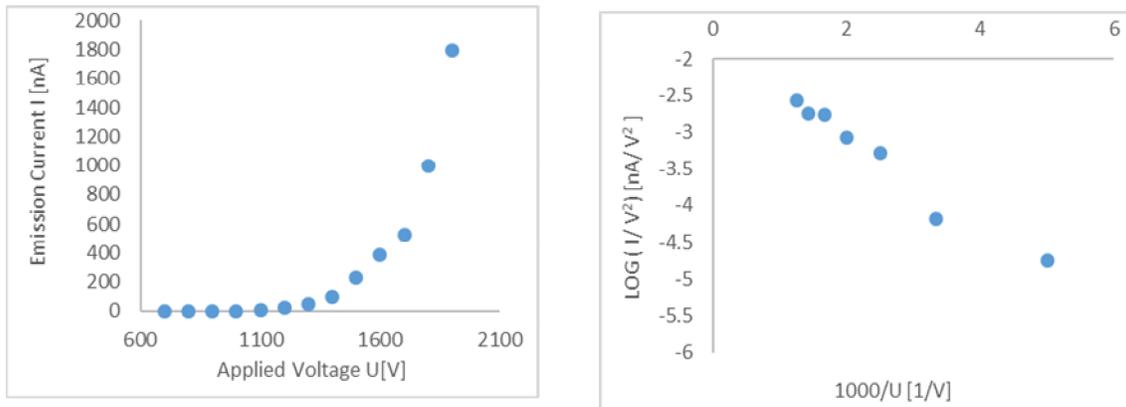


FIG. 3. The I-V characteristics (left) of composite Tungsten-Clark Electromedical Instruments Epoxylite resin during the second increasing voltage; and Fowler-Nordheim plot (right). The F-N plot slope is 260 decades per inverse volt. The applied voltage is in the range of (700 – 1900) V.

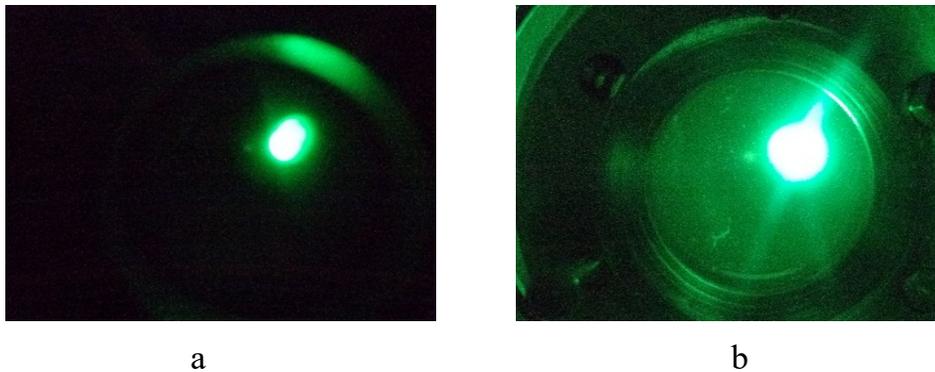


FIG. 4. Comparison between the emission of the tungsten tip before and after coating by Clark Electromedical Instruments Epoxylite resin, (a) Image of field emission of clean tungsten tip before coating. This image was taken at (1050V, 64 nA); and (b) Image of field emission for composite Tungsten-Clark Electromedical Instruments Epoxylite resin tip taken at (1700 V, 1 μ A).

Composite Tungsten - Microemitter Epidian 6 (Based on Bisphenol A) Tip

Initially presenting the characteristics of clean tungsten emitter before coating by Microemitter Epidian 6 (based on Bisphenol A), the voltage applied was in the range (390 – 1000) V and the current in the range (8 pA - 1.1 μ A). Fig. 5 (left) shows I-V characteristics, with the F-N plot on the right side of the same figure. The voltage applied on the composite Tungsten - Coated Microemitter Epidian 6 (based on Bisphenol A) tip was slowly increased across a virgin emitter until a "switch-on voltage" V_{sw} was reached. At this point, " V_{sw} ", the emission current switches

from an effective zero-value to a stable saturated value, the "switch-on current" I_{sat} . The V_{sw} is 6900V and I_{sat} is 1.2 μ A. Fig. 6 (left) shows the I-V characteristics for the first decreasing voltage and switch-on voltage with voltage range (6900 - 500) V and current range (1.2 μ A - 8.3 pA), with the F-N plot on the right side of the same figure. Fig. 7 (left) shows the voltage increasing range (1100- 2900) V and current range (11 pA - 1 μ A), with the F-N plot on the right side of the same figure. Fig. 8 shows a comparison between the field emission of the tungsten tip before and after coating by Microemitter Epidian 6 (based on Bisphenol A).

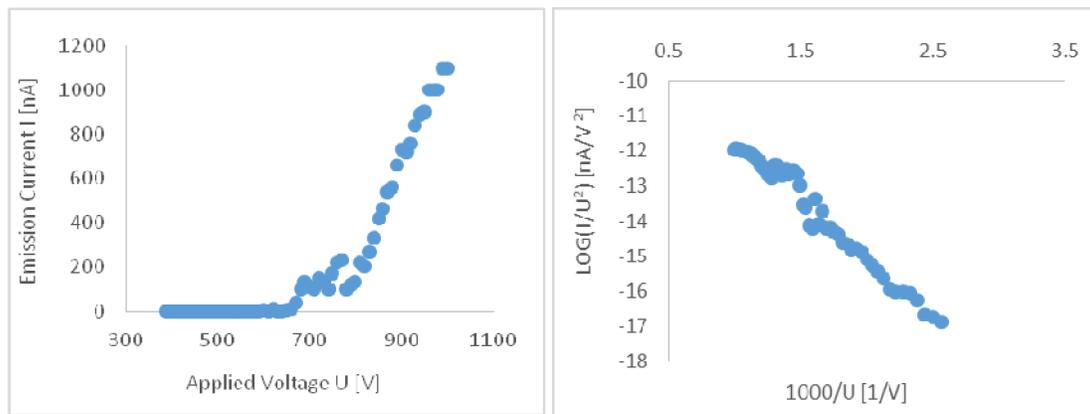


FIG. 5. The I-V characteristics (left) of clean tungsten tip whilst increasing the voltage before coating by Epidian 6 (based on Bisphenol A); and Fowler-Nordheim plot (right) with a slope of 3400 decades per inverse volt. The applied potential is in the range of (390 - 1000) V.

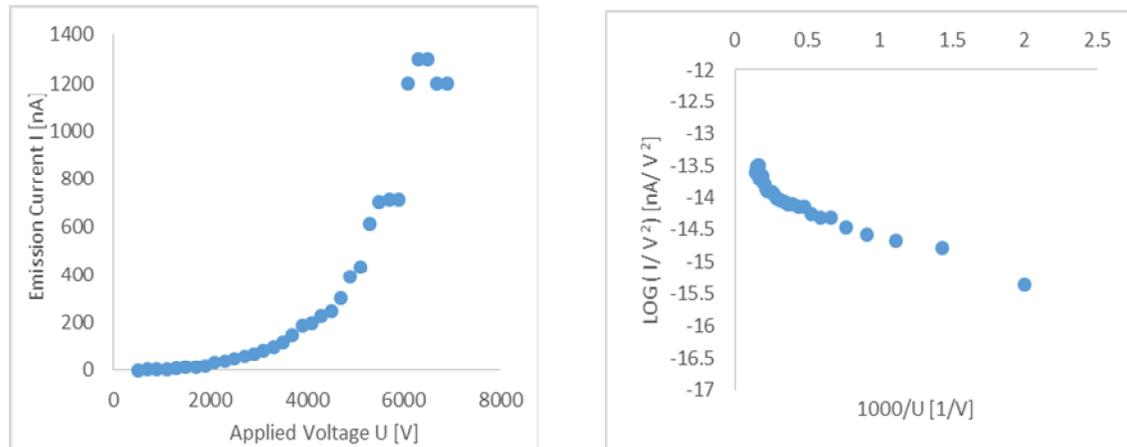


FIG. 6. The I-V characteristics (left) of composite Tungsten - Epidian 6 (based on Bisphenol A) during the first decreasing voltage with V_{sw} (6900 V, 1.2 μ A); and Fowler-Nordheim plot (right). The F-N plot slope is 950 decades per inverse volt. The applied voltage is in the range of (6900 – 500) V.

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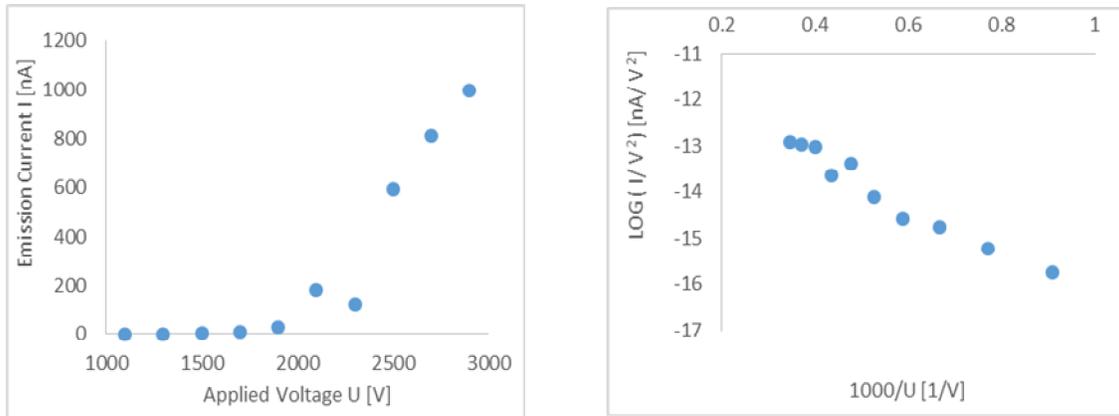


FIG. 7. The I-V characteristics (left) of composite Tungsten - Epidian 6 (based on Bisphenol A) during the second increasing voltage; and Fowler-Nordheim plot (right). The F-N plot slope is 5300 decades per inverse volt. The applied voltage is in the range of (1100 – 2900) V.

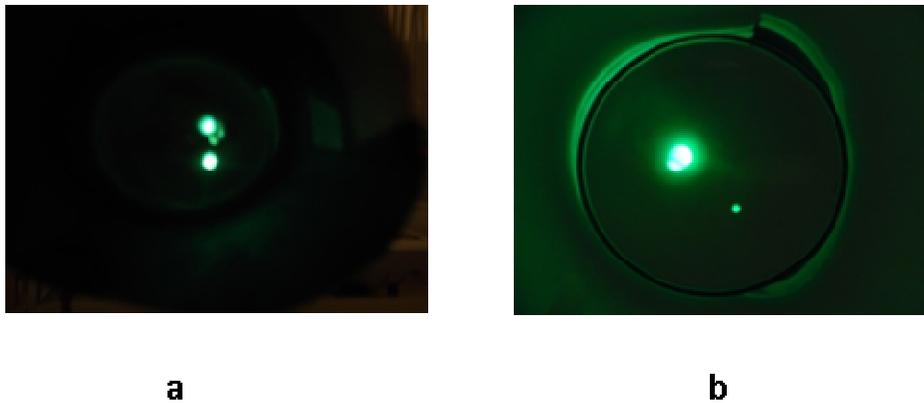


FIG. 8. Comparison between the emission of the tungsten tip before and after coating by Microemitter Epidian 6 (based on Bisphenol A), (a) Image of field emission of clean tungsten tip before coating. This image was taken at (1050V, 64 nA); and (b) Image of field emission for composite Tungsten - Microemitter Epidian 6 (based on Bisphenol A) tip taken at (1700 V, 1 μ A).

Composite Tungsten - A Radianox Compound of Graphite Colloid Tip

Presenting the characteristics of clean tungsten emitter before coating by A Radianox compound of graphite colloid, the voltage applied was in the range (800 – 1800) V and the current in the range (76 pA - 770 nA). Fig. 9 (left) shows I-V characteristics, with the F-N plot on the right side of the same figure. The voltage applied on the Composite Tungsten - A Radianox compound of graphite colloid tip was slowly increased across a virgin emitter until a "switch-on voltage" V_{sw} was reached. At this point, " V_{sw} ", the emission current switches on

from an effective zero-value to a stable saturated value, the "switch-on current" I_{sat} . The V_{sw} is 2300 V and I_{sat} is 5.4 μ A. Fig. 10 (left) shows the I-V characteristics for the first decreasing voltage and switch-on voltage with voltage range (2300- 600) V and current range (5.4 μ A - 9.1 pA), with the F-N plot on the right side of the same figure. Fig. 11 (left) shows the voltage increasing range (400- 1300) V and current range (11 pA - 780 nA), with the F-N plot on the right side of the same figure. Fig. 12 shows a comparison between the field emission of the tungsten tip before and after coating by A Radianox compound of graphite colloid.

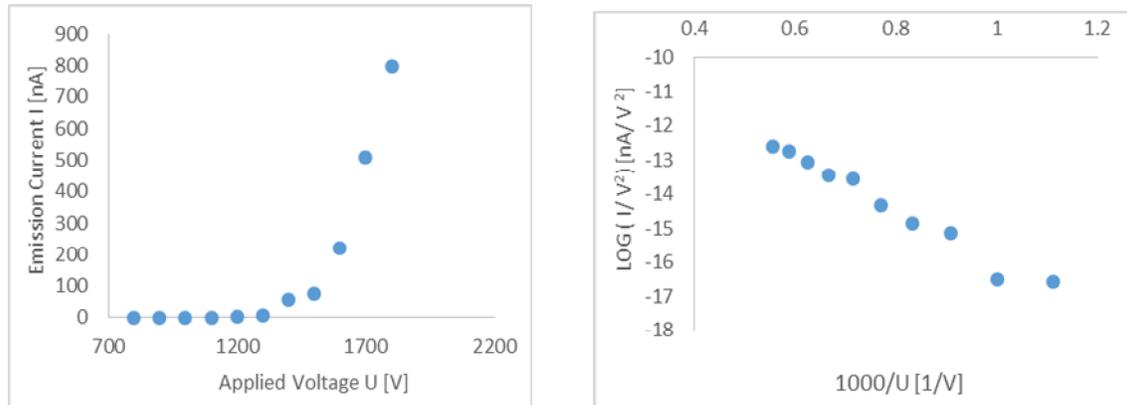


FIG. 9. The I-V characteristics (left) of clean tungsten tip whilst increasing the voltage before coating by A Radianox compound of graphite colloid; and Fowler-Nordheim plot (right) with a slope of 6800 decades per inverse volt. The applied voltage is in the range of (800 – 1800) V.

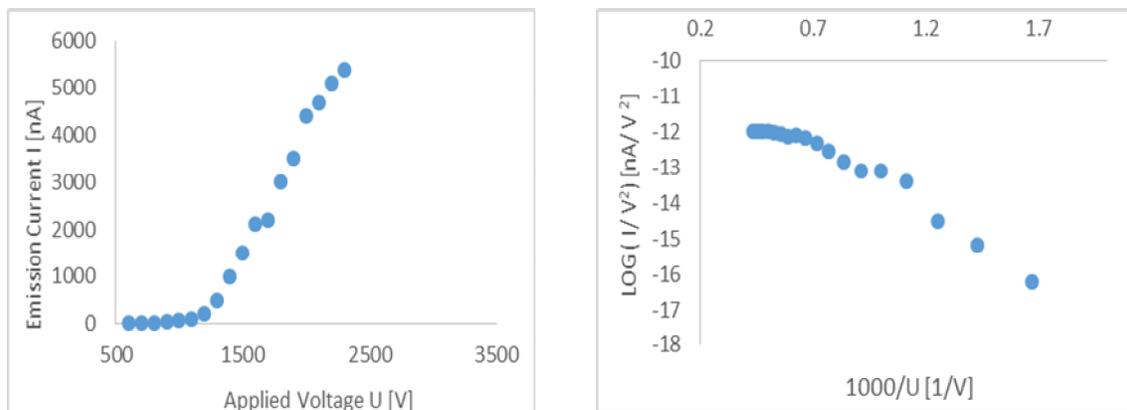


FIG. 10. The I-V characteristics (left) of composite Tungsten - A Radianox compound of graphite colloid during the first decreasing voltage with V_{sw} (2300 V, 5.4 μ A); and Fowler-Nordheim plot (right). The F-N plot slope is 3400 decades per inverse volt. The applied voltage is in the range of (2300 – 600) V.

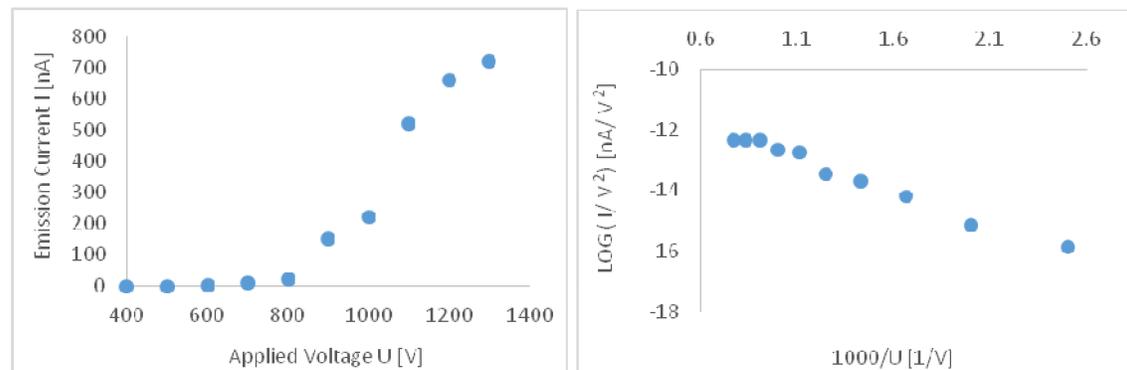


FIG. 11. The I-V characteristics (left) of composite Tungsten - A Radianox compound of graphite colloid during the second increasing voltage; and Fowler-Nordheim plot (right). The F-N plot slope is 2200 decades per inverse volt. The applied voltage is in the range of (400 – 1300) V.



FIG. 12. Comparison between the emission of the tungsten tip before and after coating by A Radianox compound of graphite colloid, (a) Image of field emission of clean tungsten tip before coating. This image was taken at (1800 V, 0.8 μ A); and (b) Image of field emission for composite Tungsten - A Radianox compound of graphite colloid tip taken at (2100 V, 4.7 μ A).

Composite Tungsten - Molyslip 2001 E Compound (MoS₂ and MoS) Tip

Presenting the characteristics of clean tungsten emitter before coating by Molyslip 2001 E compound (MoS₂ and MoS), the voltage applied was in the range (1100 – 2100) V and the current in the range (66 pA - 520 nA). Fig. 13 (left) shows I-V characteristics, with the F-N plot on the right side of the same figure. The voltage applied on the Composite Tungsten - Molyslip 2001 E compound (MoS₂ and MoS) tip was slowly increased across a virgin emitter until a "switch-on voltage" V_{sw} was reached. At this point, " V_{sw} ", the emission current switches on

from an effective zero-value to a stable saturated value, the "switch-on current" I_{sat} . The V_{sw} is 2500 V and I_{sat} is 7.3 μ A. Fig. 14 (left) shows the I-V characteristics for the first decreasing voltage and switch-on voltage with voltage range (2500 - 600) V and current range (7.3 μ A - 88 pA), with the F-N plot on the right side of the same figure. Fig. 15 (left) shows the voltage increasing range (500 - 1300) V and current range (11 pA to 440 nA), with the F-N plot on the right side of the same figure. Fig. 16 shows a comparison between the field emission of the tungsten tip before and after coating by Molyslip 2001 E compound (MoS₂ and MoS).

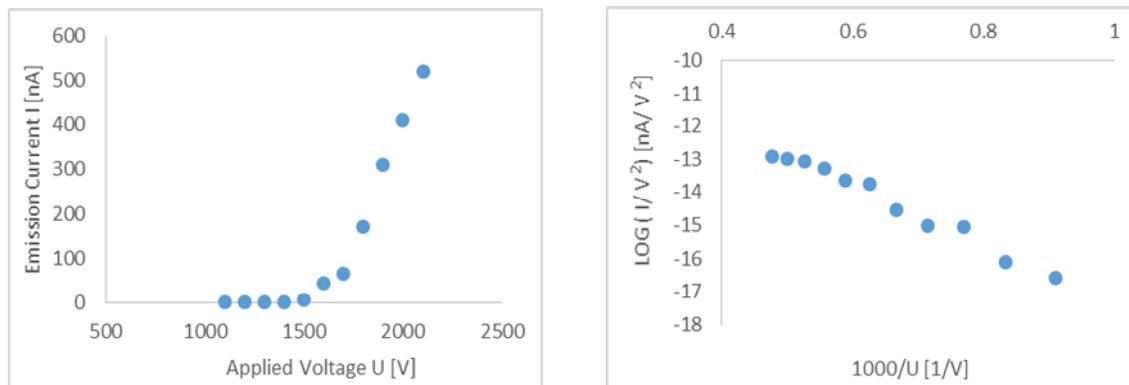


FIG. 13. The I-V characteristics (left) of clean tungsten tip before coating by Molyslip 2001 E compound (MoS₂ and MoS) whilst increasing the voltage; and Fowler-Nordheim plot (right). The F-N plot slope is 9000 decades per inverse volt. The applied voltage is in the range of (1100 – 2100) V.

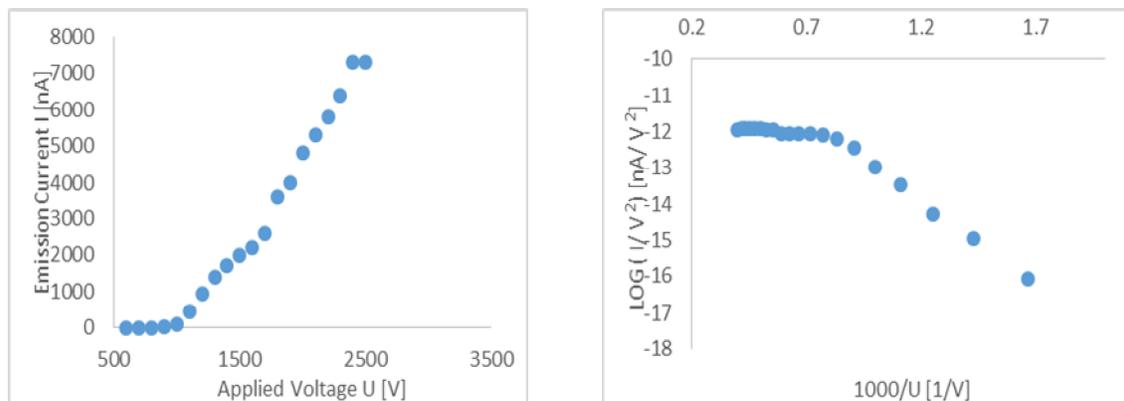


FIG. 14. The I-V characteristics (left) of composite Tungsten - Molyslip 2001 E compound (MoS_2 and MoS) during the first decreasing voltage with V_{sw} (2500 V, 7.3 μA); and Fowler-Nordheim plot (right). The F-N plot slope is 3100 decades per inverse volt. The applied voltage is in the range of (2500 – 600) V.

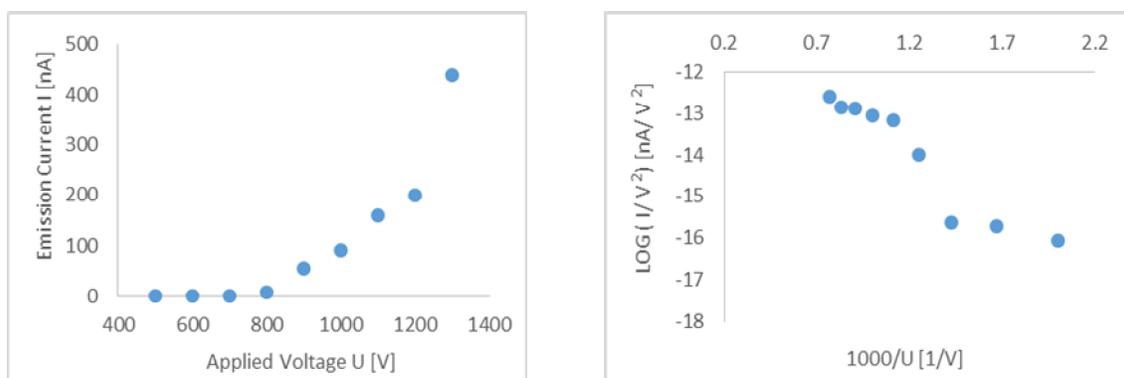


FIG. 15. The I-V characteristics (left) of composite Tungsten - Molyslip 2001 E compound (MoS_2 and MoS) during the second increasing voltage; and Fowler-Nordheim plot (right). The F-N plot slope is 3300 decades per inverse volt. The applied voltage is in the range of (500 – 1300) V.

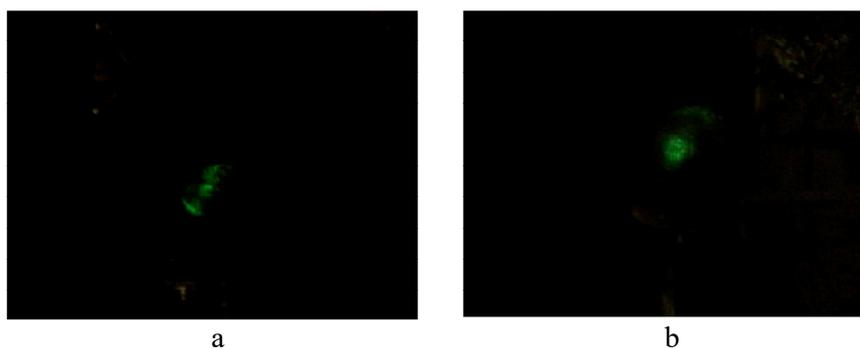


FIG. 16. comparison between the emission of the tungsten tip before and after coating by Molyslip 2001 E Compound (MoS_2 and MoS), (a) Image of field emission of clean tungsten tip before coated. This images was taken at (2100 V, 0.5 μA); and (b) Image of field emission for composite Tungsten - A Molyslip 2001 E Compound (MoS_2 and MoS) tip taken at (1300 V, 0.4 μA).

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TABLE 1. The extent of changes on the current-voltage (I-V) characteristics of the modified composite electron emitters after being coated with dielectric layer.

Emitter	Clean tip characteristics			Composite tip Characteristics					
	Applied Voltage Range (V)	Emission Current Range (nA)	F-N plot Slope (decades per inverse volt)	Switch-on Applied Voltage Range (V)	Switch-on Emission Current Range (nA)	F-N plot Slope (decades per inverse volt)	Applied Voltage Range (V)	Emission current range (nA)	F-N plot Slope (decades per inverse volt)
Composite Tungsten – Clark Electromedical Instruments Epoxylite resin	660 - 1230	0.0011- 1500	5500	2400 - 700	1500 – 0.0088	3800	700 - 1900	0.0067 - 1100	260
Composite Tungsten – Microemitters Epidian 6 (based on Bisphenol A)	390 - 1000	0.008 - 1100	3400	6900 - 500	1200 – 0.0083	950	1100 - 2900	0.0011 - 1000	5300
Composite Tungsten – A Radianox Compound of Graphite Colloid	800 - 1800	0.0076 - 770	6800	2300 - 600	5400 – 0.0091	3400	400 - 1300	0.0011 - 780	2200
Composite Tungsten - Molyslip 2001 E Compound (MoS ₂ and MoS)	1100 - 2100	0.0066 - 520	9000	2500 - 600	7300 – 0.0088	3100	500 - 1300	0.0011 - 440	3300

Discussion and Conclusions

In composite emitters consisting of clean tungsten tips with known profile, coated with a variety of insulating materials, the field emission characteristics of a tungsten electron source are intrinsically changed by coating the tips with a micro-thin layer of insulating material. This is in line with the results obtained from similar studies [12-15]. This change in characteristics varies depending on the type of insulating material used in coating the tungsten tips; this is evident by comparing the effects of insulation materials used in this work (Clark Electromedical Instruments EpoxyLite resin, Epidian 6 (based on Bisphenol A), A Radianox

compound of graphite colloid and Molyslip 2001 E compound (MoS₂ and MoS)) on tungsten tip properties. According to the results, A Radianox compound of graphite colloid and Molyslip 2001 E compound (MoS₂ and MoS) have shown an improvement in I-V characteristics and were consequently getting on higher current at the same voltage. Moreover, the emission current during using the four isolators becomes more focused and brighter. Table 1 shows the extent of changes on the I-V characteristics of the modified composite electron emitters after being coated with dielectric layer.

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