

## Experimental investigation of field electron emission from uncoated and coated graphite fiber tips

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This study investigates the field emission characteristics of coated and uncoated graphite fiber tips under high vacuum conditions in the pressure range of  $10^{-6}$  Pa. A 2 M Sodium hydroxide (NaOH) solution was used in the electrochemical etching procedure to produce an uncoated etched graphite fiber emitter with a diameter of 436.5 nm. An epoxy coating 2301 dielectric material with a thickness of  $61 \pm 1$  nm was used to coat the fiber tip. Field-emitter emission micrographs, current stability, and current-voltage (*I-V*) characteristics were recorded. Murphy's Good plots were used to examine and analyse the *I-V* characteristics. An orthodoxy test was performed to evaluate the Murphy Good plots. In the reported results, at low applied voltages, the uncoated graphite fiber emitter passed orthodoxy tests, and generated a steady current with 0.03  $\mu$ A uncertainty. However, the coated graphite fiber emitter showed an increase in the applied voltage and threshold voltages but had no impact on the sample emission characteristics.

Keywords: graphite fibre, field electron emission, epoxy resin, electrochemical etching, orthodoxy test

### 1 Introduction

Electron emission is an essential and basic phenomenon in modern science and a contemporary subject of technology, such as cathode ray tubes. Cold-field electron emission (CFE) is a type of electron emission in which a high emissive current is obtained, and is useful in many electronic applications and modern electronic circuits [1-3].

A significant element influencing the performance of electron microscopy is tip quality. This quality depends on the size, sharpness, shape, and surface cleanliness of the tip [4-6]. The most common material used as an electron source is tungsten [7, 8]. This is because of their high melting temperatures, high mechanical strengths, and relatively easy preparation by applying the well-known electrochemical etching technique [8]. Currently, ultra-sharp tips with diameters less than 90 nm are mainly used in electron guns with cathodes that operate at room temperature [8]. This type of electron source has been shown to have the highest source brightness combined with the lowest energy spread compared with other common emitters.

Graphite fiber has attracted considerable attention as a potential candidate for CFE source emitters owing to its distinct properties [9, 10]. These properties include low cost, good electrical conductivity, thermal

conductivity, and thermoelectric power within the temperature range of 10-370 K [10]. Heremans and Beetz [10] revealed that heat treatment significantly enhanced the thermal conductivity, increasing it by a factor of 50. It was noted that highly heat-treated fibers exhibited exceptional thermal conductivity, ranking among the top thermal conductors at room temperature. The low-temperature thermal conductivity data were quantitatively analysed using the phonon dispersion relation for graphite, which generally exhibits high tensile and compressive strengths, excellent stress properties, does not corrode, undergoes heat treatments above 3000 °C, and has a three-dimensional arrangement. At a heat treatment temperature of 3000 °C, the dependence of the thermoelectric power on room temperature approaches that of single-crystal graphite, indicating a high degree of crystallographic perfection of the fiber [11]. Forbes [12] comprehensively reviewed the growing importance of carbon films and carbon-based materials in scientific and technological applications.

The main objective of this study was to investigate the field-emission characteristics of coated and uncoated graphite fiber tips under ultra-high vacuum conditions. The study of emitter properties is based on the analysis of the current-voltage (*I-V*) characteristics. Fowler-Nordheim (FN) Murphy Good (MG) plots were used to

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analyse the  $I$ - $V$  characteristics associated with cold field electron emission (CFE) [13]. The slope and intercept correction factors of these plots were used to quantitatively extract more reliable emitter parameter values. These are the voltage conversion length ( $\zeta_C$ ) and emission area ( $A_f^{SN}$ ).  $\zeta_C$  can be obtained from the line slope, and  $A_f^{SN}$  can be obtained from the intercept value between the line and vertical axis. The difference between the MG and FN plots is the Kapa ( $k$ ) factor, which appears in the  $I$ - $V$  equation (1).  $k$  value in the FN equation is always equals to two. However, in the MG plots, it depends on the work function ( $\phi$ ) of the material used to produce the field-emission tip. In this study, the MG plots were used because they are more appropriate and accurate than FN plots [13].

By applying the MG plots, the relationship between the measured current  $I_m$  and voltage  $V_m$  for a single tip can be expressed as follows [13]:

$$I_m = A_f^{SN} \theta(\phi) \exp[\eta(\phi)] V_{mR}^k V_m^k \exp\left[-\frac{\eta V_{mR}}{V_m}\right] \quad (1)$$

Here,  $A_f^{SN}$  is the formal emission area through the Schottky-Nordheim potential barrier and  $\theta(\phi)$  and  $\eta(\phi)$  are functions of the work function  $\phi$ . These can be expressed as follows:

$$\theta(\phi) = a c_s^{-4} \phi^3 \quad (2)$$

$$\eta(\phi) = b c_s^2 \phi^{0.5} \quad (3)$$

where  $a$  and  $b$  are the first and second Fowler-Nordheim constants, respectively, and  $c_s$  is the Schottky constant potential barrier, which can be expressed as:

$$c_s = e^3 / 4\pi\epsilon \quad (4)$$

$V_{mR}$  is the reference measured voltage and can be expressed as follows:

$$V_{mR} = F_R \xi_C \quad (5)$$

where  $\xi_C$  is the voltage conversion length and  $F_R$  is the reference field required to drop the potential barrier to zero.

$$F_R = c_s^{-2} \phi^{-2} \quad (6)$$

where  $\kappa$  is the exponential factor of the MG plot. If only the barrier effects are considered,  $\kappa$  is given by

$$\kappa = 2 - \eta(\phi)/6 \quad (7)$$

The work function of the graphite fibre was 4.5 eV [14] and  $\kappa$  was 1.227.

## 2 Tip material and preparation method

### 2.1 Materials

To prepare the graphite fiber emitter, a fine graphite fiber wire with a purity of 99.5% was used from (Alfa Aesar). The wire had a diameter of 8  $\mu\text{m}$  and 25.4

mm long. A solution of sodium hydroxide (NaOH) at a concentration of 2 Molar was used as an etchant.

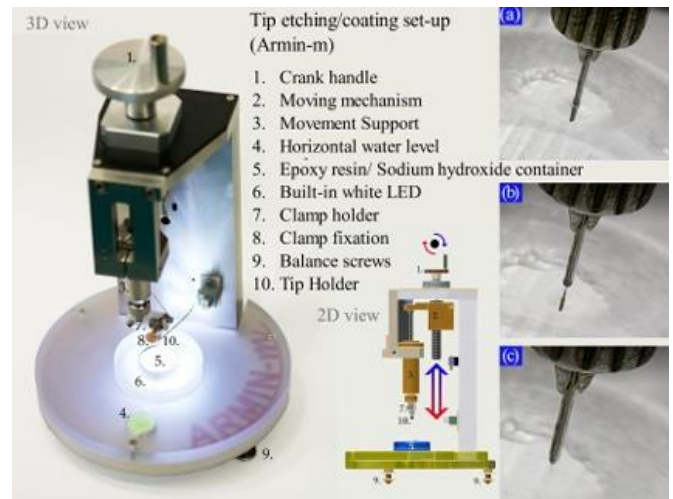
Graphite fiber is a delicate material that is challenging to control. It is sensitive to the lighting of the magnifying lens because it can be pulled by static electricity. The working environment during sample preparation must be isolated to prevent it from affecting emitter creation [15]. In addition, graphite fiber dust may cause eye irritation.

An epoxy coating 2301 dielectric material from Resinpal-Germany was used to coat the graphite fiber emitter. In previous studies [16-18], an epoxy coating on the tip was shown to provide hardness and strength. Generally, the epoxy coating layer improves the ( $I$ - $V$ ) characteristics, stabilizes the current emission, extends the tip lifetime, and increases the current densities at lower operating voltages. In addition to these benefits, the tip can be operated at lower vacuum levels.

In this study, the graphite fibers were cut to a length of 2 cm. The graphite fiber was then placed inside a stainless-steel tube that was cut to a length of 1.5 cm. A small amount of silver conductive paint was used at both ends of the tube to fix it on one side and prevent it from exiting the other end.

### 2.2 Tip preparation method

In this study, a drop-off electrochemical etching technique was employed to fabricate a graphite tip, as shown in Fig. 1 [19, 20].



**Fig. 1.** Configuration for the electrolytic etching machine [19, 20]

This method provides the possibility of reproducing the shape and sharpness of a tip. The type and concentration of the hydroxide solution allow con-

trolling the sharpness and quality of the sharpness of the tip. In this study, the etching solution prepared by using a 2 M sodium hydroxide (NaOH) solution. Generally, the metal ring and graphite fiber are the two electrodes, and the voltage between them is 12 V with direct current (DC). This technique enables the graphite sample to be held perpendicular to the electrolyte surface, as shown in Fig. 2. 2D view. Subsequently, the sample was immersed in the etching solution, where the immersed part should not exceed a length of 2 mm, as shown in Fig. 1(a), and surface tension was used to etch the graphite fibers, as shown in Fig. 1(b). The occurrence of etching was inferred from the sudden drop in the current when monitored using a multimeter. Subsequently, the prepared tip was withdrawn very quickly and vertically from the solution to prevent further etching of the tip and loss of its apex, Fig. 1(c). The etching process took less than 5 min. After the etching process, the tip was immersed for 15 to 30 min in a bowl of distilled water within an ultrasonic unit to remove any residual hydroxide solution.

### 2.3 Coating process

To prepare the coating layer, Resinpal 2301 epoxy resin was mixed homogeneously in the proportion of 100 parts epoxy resin and 50 parts epoxy hardener (by weight). The laminate was cured for 1 hour before use. The coating mechanism is carried out using the etching device. This was achieved by replacing the container of the NaOH solution with another container filled with epoxy resin. The etching device was used to ensure the accuracy of the dipping process and ensure that the angle between the sample and the epoxy surface was 90°. A coated graphite fiber tip was prepared by dipping the tip once into an epoxy resin container. The prepared coated tip was left for 24 hours until the resin has been hardened on the tip.

### 2.4 Field emission microscope (FEM) system

A field emission microscope (FEM) was used to investigate the current-voltage characteristics of the prepared graphite fiber tips (Fig. 2). The FEM operates under ultrahigh-vacuum (UHV) conditions. It provided a vacuum of up to  $10^{-6}$  Pa. The tested tip (cathode) was placed approximately 1 cm from the phosphorus screen (anode). The tip was connected to a negative high-voltage power supply (FUG HCN140-6500) via a high safety resistor (100 M $\Omega$ ), whereas the phosphorus screen was grounded and connected to a Keithley 485 pico-ampere meter. This ultrahigh vacuum is necessary to reduce the rate at which ionised gas molecules bombard the tip (cathode), thereby increasing its operational lifetime and protecting it from damage. An electron

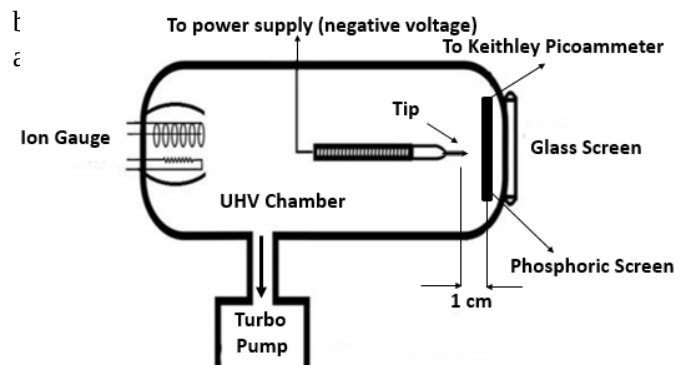


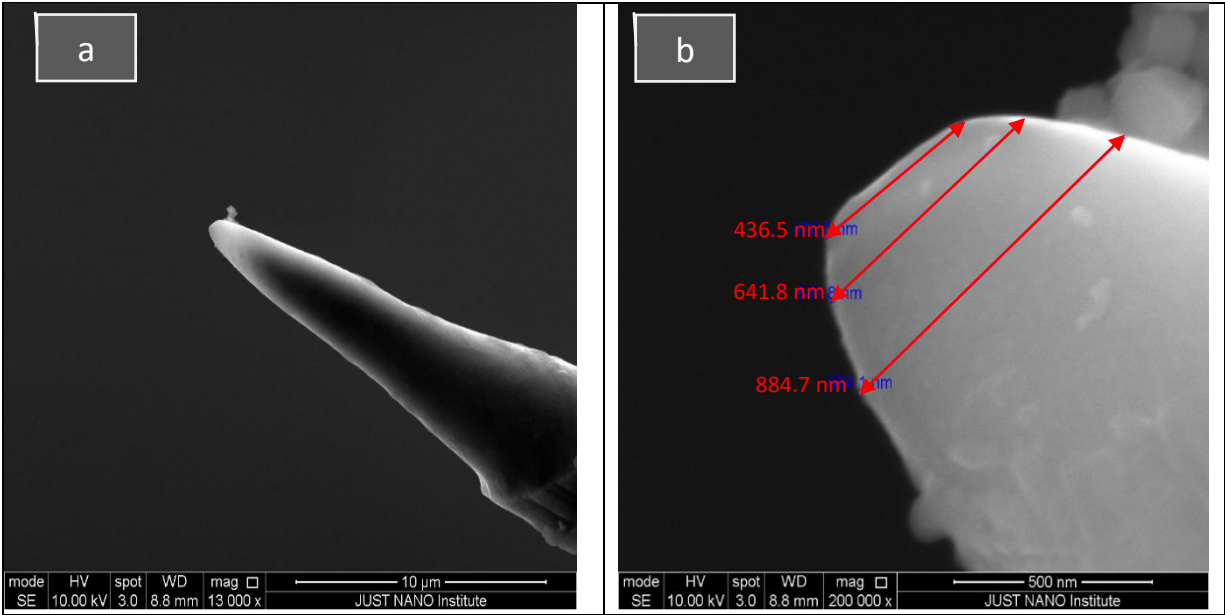
Fig. 2. Schematic diagram of the field emission microscope (FEM) set

## 3 Results and discussion

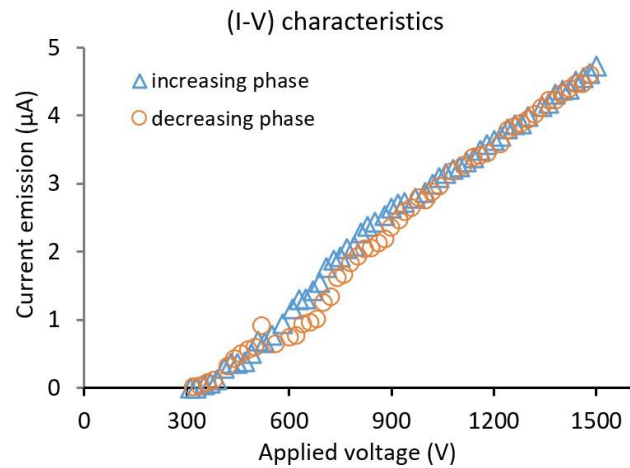
### 3.1 Graphite fibre sample (uncoated tip) ( $G_1$ )

To measure the diameter of the graphene fiber tip, scanning electron microscopy branded by FEI Quanta 450 FEG with an accuracy of 1 nm at 30 keV was used. The SEM images of the tip recorded at various magnifications are shown in Fig. 3. From these images, the tip diameter was determined to be approximately  $436.5 \pm 0.5$  nm. It was clear that the tip apex was inconsistent. This may be because the carbon fiber rod was not properly placed vertically during the etching process [19].

The measurement current-voltage I-V characteristics of the cathode for  $G_1$  over a full applied voltage cycle were measured using an ultra-high-vacuum FEM. The voltage cycle consists of two phases: increasing (triangular symbols) and decreasing (circular symbols). The increasing phase began at a certain voltage when the emission current was in the picoampere (pA) range, and the applied voltage increased periodically until the current reached 1 A. However, the decreasing phase was considered when the applied voltage began to decline periodically until the current values returned to the pA levels. Voltage was applied for multiple cycles to remove any contamination from the apex surface of the sample. To achieve a formal emission process from the surface of the graphite fibre, three cycles were completed until the switch-on procedure for the current dissipated. During the third cycle of the experiment, the current-voltage data were recorded for the two phases, as shown in Fig. 4. The current emission started at 310 V, with a current of 4.70 pA. In addition, Fig. 4 shows that satisfactory emission behaviour (2.87  $\mu$ A) was observed at 1000 V and that the turn-on voltage was 330 V at 2.52 nA. In contrast, for the 120 nm radius W tip, the emission current was 3 nA at a turn-on voltage of 700 [7]. Furthermore, the emission current reached 2.5  $\mu$ A at an applied voltage of 1000 V. Based on this information, it can be concluded that graphite fibers can be operated at a lower or the same applied voltage as W.



**Fig. 3.** SEM images of a graphite fiber sample prepared by electrochemical etching with NaOH solution with different magnifications: a) 13000× and b) 200000×



**Fig. 4.** Experimental current-voltage *I-V* characteristics of uncoated graphite fiber sample (G1) measured under a high vacuum FEM

**Table 1.** The increasing and decreasing parts of a full cycle of the applied voltage for G1

|                 |                 |         |          |         |         |
|-----------------|-----------------|---------|----------|---------|---------|
| Full cycle      | Applied voltage | 330 V   | 590 V    | 1000 V  | 1300 V  |
| Increasing part | Current         | 2.52 nA | 0.990 μA | 2.87 μA | 3.99 μA |
| Decreasing part | Current         | 20.5 nA | 0.746 μA | 2.76 μA | 3.95 μA |

**Table 2.** Field emission orthodoxy test and field emission analysis results for uncoated graphite Fiber  $G_1$ 

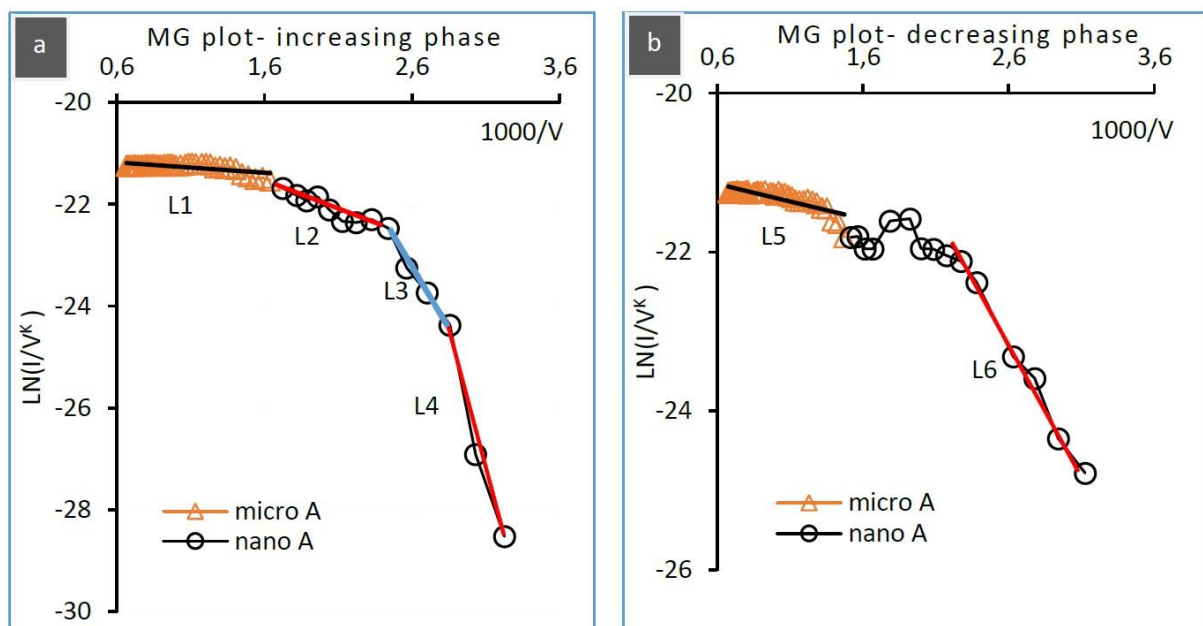
| Operating cycle | Line segments  | Slope (NP.V) | Lower operating voltage (V) | Higher operating voltage (V) | $f_{low}^{extr}$ | $f_{up}^{extr}$ | Orthodoxy test result | Voltage conversion length ( $\zeta_c$ ) (m) | Actual emission area $A_f^{SN}$ (m <sup>2</sup> ) |
|-----------------|----------------|--------------|-----------------------------|------------------------------|------------------|-----------------|-----------------------|---------------------------------------------|---------------------------------------------------|
| Increasing      | L <sub>1</sub> | -0.42        | 580                         | 1500                         | 6.42             | 16.6            | Fail                  | -                                           | -                                                 |
| Increasing      | L <sub>2</sub> | -1.65        | 410                         | 550                          | 1.32             | 1.55            | Fail                  | -                                           | -                                                 |
| Increasing      | L <sub>3</sub> | -7.73        | 350                         | 390                          | 0.19             | 0.25            | Pass                  | $1.19 \times 10^{-10}$                      | $7.12 \times 10^{-18}$                            |
| Increasing      | L <sub>4</sub> | -11.3        | 310                         | 350                          | 0.13             | 0.14            | Inconclusive          | $1.73 \times 10^{-10}$                      | $1.03 \times 10^{-12}$                            |
| Decreasing      | L <sub>5</sub> | -0.68        | 660                         | 1500                         | 4.53             | 10.2            | Fail                  | -                                           | -                                                 |
| Decreasing      | L <sub>6</sub> | -3.12        | 320                         | 440                          | 0.48             | 0.65            | Inconclusive          | $4.79 \times 10^{-11}$                      | $2.64 \times 10^{-23}$                            |

Table 1 shows the  $I$ - $V$  data for  $G_1$  for the increasing and decreasing phases of the entire cycle. It can be observed that the graphite fiber did not exhibit repeatable performance under low operating conditions. However, they exhibited repeatable characteristics at high operating energies.

Figure 5 shows the MG plots for  $G_1$ 's full cycle which takes the form  $\ln(1/V^2)$  versus  $1000/V$ . Each curve of the plots was divided into two parts: the first part was recorded when the current value reached the microampere range (triangular symbol) and the second part when the current was in the nano-ampere range (circle symbol). Each MG graph in Fig. 5 was divided into several straight-line segments for the orthodoxy test [21]. The discontinuity between L5 and L6 is due to the charging

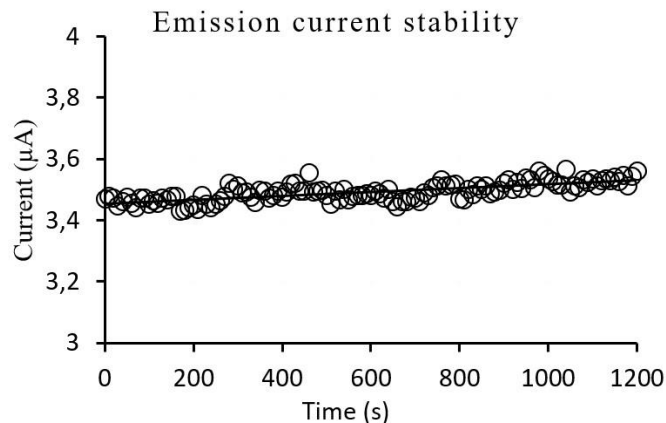
and discharging effects during the decreased cycle in the applied voltage.

The orthodoxy test is a web tool for analysing field emission results [22, 23]. The test determined and verified the validity of the prepared tips to operate as field emission tips. The slopes of these line segments are useful for extracting emission characterisation parameters such as the voltage conversion length ( $\zeta_c$ ) and actual emission area ( $A_f^{SN}$ ). This was achieved by checking whether the experiment was operated in an orthodox field emission regime. This is done by comparing the characteristic scaled field ( $f_c^{extr}$ ) of obtained data with a set of experimental reference-scaled field values ( $f_c^{ref}$ ) that have been extracted and analysed by Forbes [24].

**Fig. 5.** Murphy Good plots: (a) increasing phase and (b) decreasing phase for  $G_1$  tip

In the case of the uncoated graphite fiber sample ( $G_1$ ), the lower limit  $f_C^{low}$  is 0.15 and the upper limit  $f_C^{up}$  is 0.75. Table 2 lists the orthodoxy test and extraction results. The test was applied to the straight lines of the MG plots. The operating voltage range for each straight line is listed in Table 2.  $G_1$  passed the test for the increasing phase at low voltages between 350–390 V and was inconclusive in the voltage range 310–350 V. However, it failed at high voltages. For the decreasing cycle, the result was inconclusive in the low voltage range of 320–440 V. This could be due to several geometrical and chemical changes at the apex surface of the sample. Therefore, further studies and analyses are required to understand the exact situation of the experiment [25]. Most importantly, the actual emission areas were obtained from the MG plots. It is clear that the emission area for the increasing part of the cycle is larger than that for the decreasing part. This could be because the changes that occurred on the sample apex surface after the full cycle reached a high operating voltage and then returned to low voltages.

The current stability of  $G_1$  is shown in Fig. 6. Figure 6 shows the measured emission current, with one reading every 10 s for a period of 20 min at an operating voltage of 1200 V. The average emission current was  $3.5 \pm 0.03$   $\mu$ A.



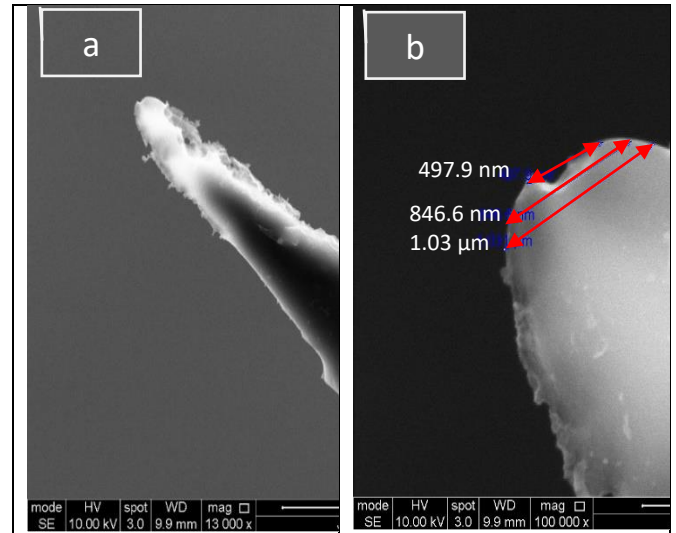
**Fig. 6.** Stabilisation of the current of the uncoated  $G_1$  tip and the changes in current over 20 minutes at a voltage of 1200 V

As a result, the uncoated carbon fibre tip exhibited a reasonable emission current. This suggests that the electrical characteristics of the graphite fibers are promising.

### 3.2 Coated $\overline{G}_1$ graphite fibre sample

A coated  $\overline{G}_1$  graphite fiber tip was prepared by dipping the  $G_1$  tip once in epoxy resin dielectric material.

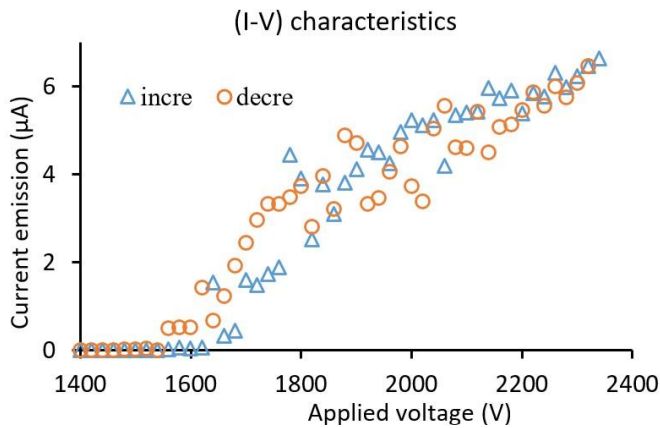
The SEM images were obtained at several magnifications, as shown in Fig. 7. The  $\overline{G}_1$  apex diameter was  $498 \pm 0.5$  nm, while the  $G_1$  apex diameter was 436.5 nm. Therefore, the thickness produced from one dip was approximately  $61 \pm 1$  nm according to the technique used to measure the distance in the SEM images.



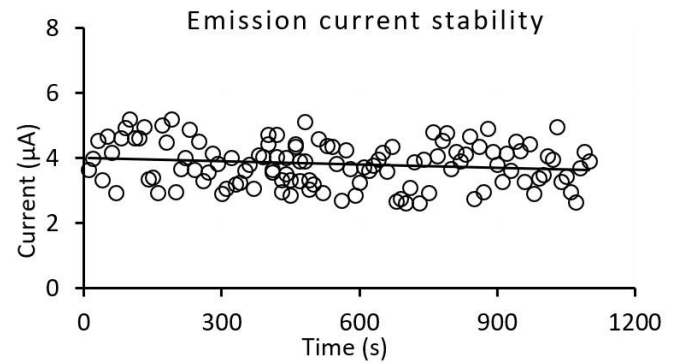
**Fig. 7.** SEM images of coated graphite fibre ( $\overline{G}_1$ ) tip at different magnifications: a) 13000 X and b)- 100000 X. The thickness of the tip of the graphite fibre  $\overline{G}_1$  approximately  $498 \pm 1$  nm.

The coated  $\overline{G}_1$  graphite fiber tip showed undesirable emission behavior, as the switch-on process did not disappear after five full recoding cycles. The data for the current-voltage characteristics of the fifth cycle in Fig. 8 show a significant increase in the threshold voltage until the actual emission current reached 8 nA has been obtained at an applied voltage of 1500 V. On the contrary, the uncoated graphite fiber tip exhibited a threshold voltage of 330 V, as shown in Fig. 4. The coating process did not result in a reduction of the applied voltage required to produce the same emission current. This could be due to the quality of the contact between the epoxy layer and graphite fiber tip. In the presence of a high-quality epoxy coating process and high applied electric fields, the electrons can escape the apex of the sample through a channel formed between the epoxy layer and the graphite fiber tip. This channel allows electrons to escape through the surface at low applied voltages [26]. Knápek et al. [27] proposed a new model for composites that uses electron sources as a system of capacitors. In this model, the switch-on phenomenon appears as a discharge effect following breakdown in the dielectric layer. During the charging process, electrons occupy the conduction band of the metal, forming an electron chamber. In the dielectric

epoxy layer, nanocapacitors are constructed from merged electric dipoles and larger capacitors are formed by merging these nanocapacitors. Finally, the two sides of the dielectric layer are connected via an electric vent, allowing electrons to be emitted from the electric chamber through the vent, resulting in an intense electron beam forming a channel that enables those electrons to pass through it and produce a switch-on current. The latter two assumptions explain the reduction in the turn-on applied voltage for the composite tip, which is not applicable to the obtained results. For the prepared composite tip, an increase in the turn-on applied voltage and variation in the emission current were observed on the obtained result, as shown in Fig. 9. This can be explained by the trap assumption and the charging and discharging effect occurring on the coated tip, as revealed by Adel et al. [28].



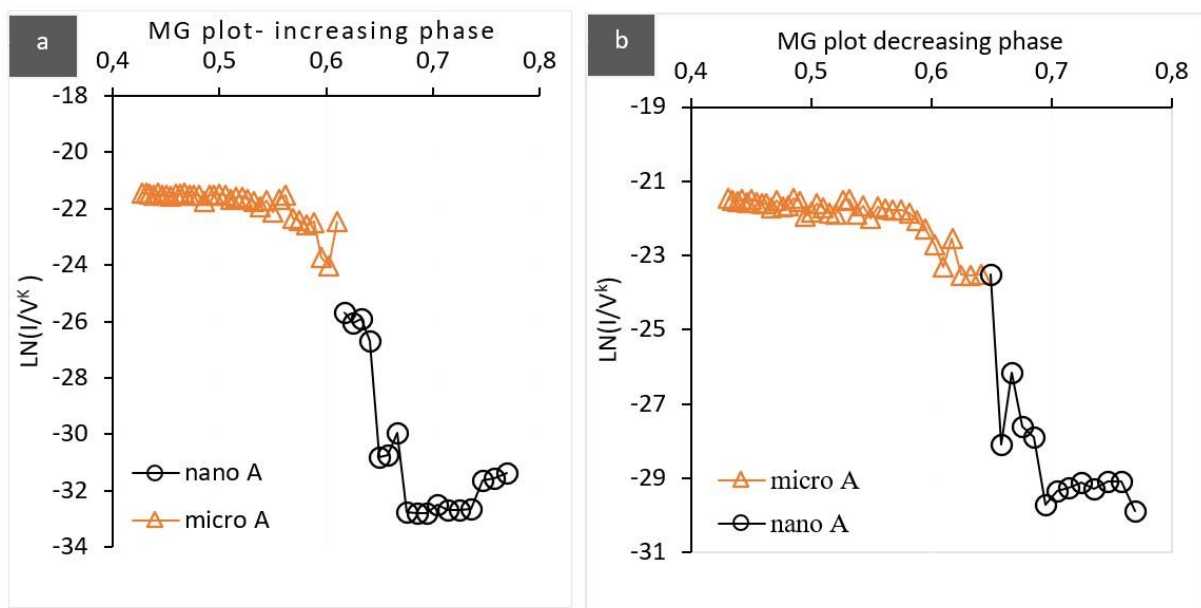
**Fig. 8.** Characteristics of the current-voltage data for coated graphite fiber  $\overline{G}_1$  sample.



**Fig. 9.** The current stabilization of  $\overline{G}_1$  tip and the changes in current over time of 20 minutes at applied voltage of 2250 V

The emission current of the inconsistent beams as a function of time was less stable than that of uncoated graphite fiber G1, as shown in Fig. 10. Figure 10 shows the measured emission current, with one reading every 10 s for a period of 20 min at an applied voltage of 2250 V. The measured recorded current fluctuated between 2.6  $\mu$ A and 5.2  $\mu$ A with an average emission current of  $3.82 \pm 0.66 \mu$ A. This fluctuation in the emission current is due to the charging and discharging effects as the voltage continues to be applied to the tip.

Figure 10 shows MG plots for  $\overline{G}_1$  over the entire cycle. Unfortunately, it is difficult to establish an even straight line for all parts that increase and decrease. Consequently, the present types of emissions are classified as thermal rather than cold-field emissions [24].



**Fig. 10.** Murphy Good plots; (a) increasing phase and (b) decreasing phase for coated graphite fibre  $\overline{G}_1$  tip

Table 3 shows the current–voltage data of  $\overline{G}_1$  for the increasing and decreasing parts of the full cycle. As can be observed, the coated graphite fiber  $\overline{G}_1$  did not produce repeatable data at low operating voltages below 1900 V.

**Table 3.** The increasing and decreasing phases of a full cycle of the applied voltage for coated graphite fiber  $\overline{G}_1$  sample

| Full cycle       | Applied voltage  | 1480 V  | 1550 V | 1700 V      | 1960 V      | 2340 V      |
|------------------|------------------|---------|--------|-------------|-------------|-------------|
| Increasing phase | Emission current | 0.48 pA | 0.5 nA | 1.5 $\mu$ A | 4.2 $\mu$ A | 6.6 $\mu$ A |
| Decreasing phase | Emission current | 1.60 nA | 1.6 nA | 2.4 $\mu$ A | 4.0 $\mu$ A | 6.5 $\mu$ A |

## 4 Conclusions

Sodium hydroxide solution was used to produce smooth and regular tips of graphite fibers, resulting in electron emission current behavior with minimal fluctuation at a low applied voltage. The obtained results for graphite fiber with 218 nm radius tip, a satisfactory emission current of 2.87  $\mu$ A was observed at 1000 V and the turn-on voltage was 330 V with a 2.52 nA emission current. In contrast, for the 120 nm radius W tip, the emission current was 3 nA at a turn-on voltage of 700. Furthermore, the emission current reached 2.5  $\mu$ A for an applied voltage of 1000 V. Based on this information, it can be concluded that graphite fibers can be operated at a lower or the same applied voltage as W with the same emission current.

The electrical and emissive characteristics of graphite fibers are often highly effective compared to those of other metal emitters, offering a higher effective emission current. These differences can be explored further in future studies to emphasize key distinctions. This comparison may be carried out in future research publications to highlight key distinctions.

The coating procedure was carried out by incorporating an insulating epoxy resin 2301 material, owing to the significance of composite materials in many technological applications such as electron microscopy. In the reported results, the sample coating resulted in an increase in the operating voltage and threshold voltages, but had no effect on the emission characteristics of the samples. The stability of the emission current was lost over time because of the coating layer.

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