A Comparative Analysis of Field Electron Emission from Carbon Black Embedded within Insulated Copper Hollowed Wires and Glass Tubes

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Abstract
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Keywords
: Carbon Black, Hollowed Insulated Copper Tip, Field emission, Field electron emission, Murphy – Good plots

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RESEARCH PAPER

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Abstract

In this study, two different methods are used to investigate carbon black as a cold field electron emitter. The first method is to incorporate carbon black into a specially designed insulated copper hollowed wire. The wire has a cup-shaped structure created by electrochemical etching. The second method involves the incorporation of carbon black into narrow glass tubes. A Comparative analyses is carried out to evaluate the effectiveness of each method. To evaluate the performance of the samples, the current—voltage characteristics will be examined using field electron microscopes. This analysis will provide an understanding of the emission of the carbon black under different conditions. Furthermore, the obtained data will be analysed and interpreted using Murphy-Good plots. Murphy-Good plots are a common graphical tool for measuring electron emission. The aim of this research is to identify the most effective and reliable techniques for the use the carbon black as a cold-field electron emitter. The research findings will contribute to the development of advanced electron emission technologies. They will also provide valuable information for the design and optimization of carbon black-based electron sources.

Keywords: Carbon black, Hollowed insulated copper tip, Field emission, Field electron emission, Murphy-Good plots

1. Introduction

Cold field electron emission phenomenon (CFE) occurs when an intense electric field is applied to the surface of metals and semiconductors. The electrons emitted from the surface pass through the potential barrier. This effect is the result of quantum mechanical tunnel phenomena [1]. The current emitted can be increased by increasing the temperature of the emitter surface by changing the distribution function (in the case of an electron, the Fermi-Dirak function) or applying strong electric fields that affect the potential barriers, called the cold electron emission produced by applied external electric fields [2]. The tunneling process takes place at energy levels close to the Fermi level and at very low temperatures in comparison to the thermionic emission of electrons. The resulting emission current \( I_m \) depends mainly on the applied measured voltage \( V_m \) between the two electrodes and the local work function \( (\phi) \) of the used material. Field emission cathodes are commonly used to produce high-resolution electron images in scanning electron microscopes (SEM) and transmission electron microscopes (TEM) [3].

Carbon black (CB) can be used as a field emitter display (FED) due to its high surface area and conductive properties. This is due its high electron
flow rate, relatively high resolution, and ability to produce detailed images of sample surfaces [4]. In additionally, Carbon Black is widely available and relatively inexpensive, making it a popular choice for many researchers and scientists. With a surface area of approximately 254 m²/g, the diameter of a single such particle ranges from 50 to 70 nm [5–8]. In reviewing the relevant literature, it has become apparent that the amount of research dealing the nuances and intricacies of pure carbon black is relatively limited. This gap underscores the importance of the proposed research in contributing to the existing knowledge base in this area. Therefore, in the present study, two emission sources were prepared using two different methods. The first method is to embed carbon black (CB) in glass tubes with an internal diameter of 0.1 mm [9]. The second method uses insulated hollow copper wires.

In general, CB dust is formed by the incomplete combustion of hydrocarbon gases and vapours from oil sources at temperatures of up to 1400 °C (with a limited supply of oxygen) [10–13].

A high conductivity carbon black manufactured by Cabot Corporation, known as Vulcan® XC-72. Vulcan® XC-72 has attracted special attention due to its good compromise between adequate surface area (~250 m²/g) and high electric conductivity (~2.77 S/cm) [14].

The aim of the present study is to evaluate the two produced emitters in order to improve the most efficient extraction mechanism of electrons from CB.

The study of the emitter properties is based on analyzing the current–voltage (I–V) characteristics. Fowler-Nordheim (F–N) and Murphy—Good (M–G) plots are usually used to analyze the (I–V) characteristics associated with cold field electron emission (CFE) [15,16]. The slope and intercept correction factors of the above (F–N) and (M–G) plots are used to extract quantitatively more reliable values of the emitter parameters. These are the field enhancement factor, the emission area, and the tip radius.

The (F–N) plots are applicable to analyse the field emission from carbon materials, as described in Forbes’ study [17]. However, in 2019 Richard G. Forbes suggested that the M–G plots are more appropriate for certain correction factors than the F–N plots [18]. The difference between M–G and F–N plots is the Kapa (k) factor which appears in the I–V equation (1). (k) value in the F–N equation is always equals to two. However in the M–G plots it depends on the work function of the material used to produce the field emission tip. Therefore, the M–G plots are used in this study to analyze the current–voltage characteristics associated with to cold field electron emission (CFE) [19].

By applying the M–G plots, the relationship between the measured current I_m and the measured voltage V_m for a single tip can be shown as follows [18]:

\[
I_m = \left\{ \frac{A_f^{SN}}{\theta} \exp(\eta V_m^{3/8}) \right\} V_m^2 \exp \left[ -\frac{\eta V_m R_{MG}}{V_m} \right]
\]  

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

\[
\theta(\phi) = a c_s^{-4} \phi^3 
\]

\[
\eta(\phi) = b c_s^2 \phi^{1/2} 
\]

\[
\kappa = 2 - \eta/6 
\]

where a and b are the first and second Fowler–Nordheim constants, \( c_s = e^β/4πε \) is the Schottky constant, and \( V_{mR} = F_R z \zeta_C \). \( F_R = c_s^2 \Phi^{-2} \) is called the reference field, which is the field required to drop the potential barrier to zero, and \( \zeta_C \) is the characteristic voltage conversion length, which is usually used to convert from measured voltage to applied electric field.

The CB surface can be treated as a Large Area Field Emitter (LAFE) [20] although the micro-emitters are not uniformly arranged [21]. Assuming that \( A_f^{SN} \) remains constant and all parameters on the right-hand side, except \( V_{mR} \) are constant, equation (1) can be predicted to have a linear relationship with a slope of \( S_{MG} \) and an intercept of \( \ln[R_{MG}] \) as following [20]:

\[
R_{MG} = \frac{A_f^{SN}}{\theta} \exp(\eta V_m^{3/8})
\]

\[
S_{MG} = -\ln R_{mR} = -b\phi^{3/2}z \zeta_C
\]

In regards to LAFEs, a property that may prove more practical is the extracted formal area efficiency \( \alpha_f^{SN} Extr \), which can be defined as the quantification of the portion of the emitter area that is actively emitting to a significant degree. Here, SN is an indication of the Schottky-Nordheim barrier, and “Extr” is an abbreviation for extraction. Extracted formal area efficiency [20]:

\[
\{\alpha_f^{SN}\} Extr = \frac{\{A_f^{SN}\} Extr}{A_M}
\]

Where the symbol \( A_M \) denotes the macroscopic area, also known as the footprint of the LAFE.
Fig. 1. SEM images of the surface pure carbon black with two different magnifications.

Fig. 2. Schematic diagram of a manual tip etching device.
2. Experimental details

2.1. Carbon black

The local work function of the material is an essential element of CFE research. Fabish & Schleifer [22] used the contact potential difference (CPD) method and found that CB Vulcan XC-72 has a work function referenced to the gold work function of about 5.58 eV with uncertainties of ±0.07 eV. Fig. 1 shows an SEM image of the CB surfaces, which clearly shows that these surfaces are characterized by many sharp nano-spikes.

Consequently, the parameters of the work function will have the following values with respect to the previously mentioned uncertainty of ±0.07 eV [18]:

\[
\begin{align*}
1.301603 &/ C20 \\
1.305998 &/ C20 \\
4.164011 &/ C20 \\
4.190378 &/ C20 \\
(1.243586 &/ C20 q)
\end{align*}
\]

and (1.291587) / C20 × 1014 A/m2.

2.2. Emitters fabrication

2.2.1. Copper insulated wire

The etching circuit, which includes two electrodes, a 60% concentrated solution and a 15 V power source, is shown in Fig. 2. A stainless steel ring around the one mole sodium hydroxide (NaOH) acts as the cathode, while a copper-insulated wire immersed in the solution acts as the anode. When the circuit is activated, the etching process begins by oxidising copper to Cu+2. The Copper dissolution can be expressed by the reaction [23].

\[
\text{Cu} + \text{H}_3\text{PO}_4 + \text{H}_2\text{O} \rightarrow (\text{CuOH})^+ (\text{H}_2\text{PO}_4)^- + 2\text{H}^+ + 2\text{e}^-
\]  

(8)

After the etching process, the wire was cleaned with distilled water using an ultrasonic cleaner. The outcome is a structure in the shape of a cup, with the front portion of the Cu wire acting as the base and the insulating material forming the lateral surface. As shown in Fig. 3a, b, and c, the next step is to fill the evacuated volume of the wire with CB soot.

The insulating material was subjected to an EDX (Energy Dispersive X-ray) analysis; Table 1 and

Table 1. An EDX (Energy Dispersive X-ray) analysis of the insulation layer indicates that the material is mainly composed of carbon and oxygen with a small amount of copper.

<table>
<thead>
<tr>
<th>Element number</th>
<th>Element symbol</th>
<th>Atomic concentration</th>
<th>Weight Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>C</td>
<td>76.45</td>
<td>60.40</td>
</tr>
<tr>
<td>8</td>
<td>O</td>
<td>18.81</td>
<td>19.80</td>
</tr>
<tr>
<td>29</td>
<td>Cu</td>
<td>4.74</td>
<td>19.80</td>
</tr>
</tbody>
</table>
Fig. 4 show the composition of this layer, which is composed mostly of carbon and oxygen with a small amount of copper.

2.2.2. Glass tips

As already mentioned, a glass tube can also be used to produce holders for emitters (tips). These glass holders were made using a device called glass puller [24], shown in Fig. 5. This device operates by heating the glass tube with a surrounding heating ring and the tube is clamped at both ends. Gravity narrows the heated part of the tube and then separates it into two sharp glass tips.

To ensure the conductivity of the CB in the experiment, the CB was inserted into the tube and pushed by a stainless-steel tube, resulting in a bulk of CB as shown in Fig. 6a and b.
Fig. 7. Optical microscope images of the samples (a) CBGlass-1, (b) CBGlass-2, (c) CBCu-1, and (d) CBCu-2.

Fig. 8. The emission pattern corresponds to the samples: (a) CBglass-1 at 6050 V with 3.1 nA, (b) CBglass-2 at 2100 V and 124 nA, (c) CBCu-1 at 4200 V with 2.8 µA, (d) CBCu-2 at 2350 V with 11.8 µA.
2.3 Field electron emission microscopy (FEM)

In this study, the Field Electron Microscope (FEM) was used to assess the samples by measuring the field electron emission current and looking at the patterns of emission current distribution on a phosphorous screen located 10 mm from the cathode (the emitter). A high accuracy pico-ampere was used to directly monitor the current as the voltage was gradually increased, with each step being 50 V. The results obtained from four samples were analyzed in this study.

3. Experimental results

In this experiment, four samples (CB-Glass-1, CB-Glass-2, CBCu-1, and CBCu-2) were evaluated and two fabrication processes were compared. Fig. 7 illustrates the four samples. The Cu-CB cathodes released currents in the tens of microamperes range, while the glass tips released currents in the nanoampere range. At room temperature, all experiments were carried out under a pressure of $10^{-6}$ mbar.

3.1 Emission from the glass tips

The sample (CB-Glass-1) showed an emission current of 0.4 nA at a voltage of 5800 V. Fig. 8a shows the spot shape of the corresponding FEM pattern image for the current distribution. Despite the application of a relatively high voltage, the resulting...
emission current was low. On the other hand, the CB-Glass-2 sample showed emission at a lower voltage of 3700 V and an emitted current of 0.031 nA. As shown in Fig. 8b, the distribution of the showed FEM pattern was spotty. Figs. 9 and 10 show the I−V characteristics and M-G analysis for the two samples.

3.2. Emission from CB-Cu tips

In comparison to the glass tube method, the production of the CBCu-1 fabrication technique resulted in a significant disparity in the emitted current. The switch-on current obtained through applying this technique was achieved at a voltage of 1850 V with an emitted current of 1.51 nA. Fig. 8c shows the emitted current pattern. Moreover, the same result was observed in the implementation of CBCu-2, where the switch-on current was achieved at a voltage of 2000 V with an emitted current of 8.02 μA. The emission pattern shown in Fig. 8d. The (I−V) characteristics and the results of the M-G analysis for the two samples are shown in Figs. 11 and 12. When this
type of emitter was replaced by glass emitters, the slope of the (I–V) characteristics was lower and the current emitted current was more steady.

The present experiments were conducted on the holder material by subjecting both the hollow copper wire and the glass tube without embedded CB material (empty) to a voltage of 6500 V each, in a FEM chamber at a pressure of 10\(^{-6}\) mbar at room temperature.

The results of the evaluation of four samples using the two different methods are shown in Table 2, which shows a significant discrepancy in the emitted current.

4. Discussion

The (CFE) of “pure CB nanoparticles” has not been thoroughly studied because prior research has
focused on CB composites with other materials such as silicon and rubber [25].

The results of the experiments conducted on the holder material, both the hollow copper wire and the glass tube without embedded CB material (empty) were subjected to a voltage of 6500 V each in an FEM chamber at a pressure of $10^{-6}$ mbar at room temperature, indicating that CB was the main source of the measured current and that the supporting materials were insignificant.

The study of the composition of the emitter, indicates that the fundamental reason for the emission current increase could be due to the variation in the quantity of CB used in each methods.

In particular, more CB is required for the glass tube approach than that for the copper hollowed wire. This is because the space between the electrically conductive metal (i.e., stainless steel in the

<table>
<thead>
<tr>
<th>Sample</th>
<th>Switch-on voltage (V)</th>
<th>Switch-on current (A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CBGlass-1</td>
<td>5850</td>
<td>$0.40 \times 10^{-9}$</td>
</tr>
<tr>
<td>CBglass-2</td>
<td>3700</td>
<td>$0.03 \times 10^{-9}$</td>
</tr>
<tr>
<td>CBCu-1</td>
<td>1850</td>
<td>$0.29 \times 10^{-9}$</td>
</tr>
<tr>
<td>CBCu-2</td>
<td>2000</td>
<td>$8.02 \times 10^{-9}$</td>
</tr>
</tbody>
</table>

Fig. 12. (a) $I-V$ plot for increasing voltage related to the sample CBglass-2: the emission started at 2000 V with an emitted current 8.02 μA. (b) Related Murphy-Good plot.
case of glass tips) and the apex of the tip contains a denser and more abundant amount of carbon material than the cup volume of copper wire. Therefore, the thickness of the carbon material appears to be related to emission efficiency.

Mousa and Latham proposed that there is a channel in the insulating material covering the surface of the metal emitters, leading to an increase in the emission concentration and the switching current [26]. Knápek et al. [27] also explained the formation and function of micro-dipoles within a very thin layer of epoxy that covers an etched polymer graphite rod that acts as the cathode. The applied voltage has a direct proportionality to the merging rate of these dipoles [26].

On the basis of this observation, a reduction in the CB layer’s thickness could result in an increase in the electron emergence rate and hence the emitted current.

5. Conclusion

A noticeable increase in the emitted current was observed when the thickness of the carbon black layer on the base of the Cu wire’s cup was varied. It is believed to be due to the fact that the micro-dipoles which were previously mentioned, can emerge much more in this case. The thick layer of material in the glass tips contains more vacancies which prevent the emergence of the micro-dipoles, increase the occurrence of secondary electrons, and cause more collisions between electrons. This could lead to higher resistance and a lower emitted current. Understanding the advantages of this electron source system and its related characteristics—such as lower threshold voltage, switch-on phenomena, and current stability—can provide important insights.

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