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ARTICLE

Investigating the Effects of Sample Conditioning on Nano-Apex Carbon Fiber Tips for Efficient Field Electron Emission

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Abstract: Carbon fibers represent a very interesting material for field electron emitters. While the preparation of sharp carbon fiber tips is very similar to the conventional method used for tungsten tips, much smaller apex radii can be obtained. Within the study presented here, such emitters have been prepared and tested using a standard field emission microscope (FEM). Various properties of these emitters were measured including the relationship between emitted electron current and applied voltage to the tip (the current-voltage characteristics) and the corresponding electron emission images. The field emission of carbon fibers has been studied via various methods of sample conditioning treatment; such as thermal relaxation process and cryogenic cooling. The performance of these tips was found to be dependent on the sample conditioning treatment.

Keywords: Electrochemical etching; Field electron emission; Fowler-Nordheim plots; Carbon fiber tips; Vacuum-based analysis.

Introduction

Field electron emission is the emission of electrons from the surface of a cathode under the influence of the applied electric field (of 3×10^9 V/ m) [1], which is strongly dependent upon the work function of the emitting material. The first explanation of what appears to be a field electron emission initiated electric discharge was made by Winkler [2].

One of the most recent studies on analyzing Fowler-Nordheim plots has been conducted by Forbes, Mousa and Fischer, which was concerned with studying the cold field electron emission [3, 4]. One of the important implementations, introduced by Al-Rabadi and Mousa, has been performed within controlled-switching (i.e., multiplexing) applications [5, 6].

The general form of Fowler-Nordheim-type (FN-type) equation is given as follows [7]:

$$J = \lambda_L \alpha \phi^{-1} F^2 \exp \left[-\frac{v_F b \phi^{\frac{3}{2}}}{F} \right]$$

This equation is used in all cases of field electron emission processes, where J is the local emission current density, a and b are the first and the second Fowler – Nordheim constants, respectively, v_F is the barrier form correction factor and it accounts for the particular shape of the potential barrier model, and λ_L is the local pre-exponential correction factor where it takes into account all of the other factors that influence the emission. ϕ is the work function of the material used. Certainly, v_F and λ_L depend on the applied field F [7].

Electron sources that are based on field emission have a number of applications that include the construction of bright electron sources for high-resolution devices and optoelectronic equipment [8].

Due to the technological importance of carbon fibers, there has been a growing interest in understanding the mechanism of the field

electron emission from these fibers under the influence of an applied electrical field [9]. Using carbon fibers as cathodes brings several benefits such as offering the ability to work in a relatively high pressure environment (about 10^{-6} mbar), simplicity of manufacturing the emitter, high current stability and long emitter durability [9]. Such cathodes are made either of carbon fibers or of other carbon-based materials.

The motivation of this work is to examine the influence of the conditioning treatment on the emitter behavior. Preparation techniques were employed to produce conical carbon fiber sharp, medium and blunt tips in order to study the means by which the emission current instability could be overcome [10]. This work aims at developing an electron source that has a long life with high emission current stability and an increased brightness. Another aim of this research is to prepare the ground to study the electron emission physical mechanism from the carbon fiber microemitters and also to be compared with that of other materials such as tungsten. It is worth noting that tungsten is the main material used in the electron emission-based technologies.

Experimental Setup

Carbon fiber emitters can be produced by electrolytic etching technique, where a 0.1 Molar of sodium hydroxide solution (250 ml of distilled water with 2g of NaOH) is used [10]. This etching process could be controlled by choosing a suitable etching current. The etching process starts after dipping the sample to be etched in the solution by about 2 mm and using a power supply to provide the dc voltage until a certain initial current of about 30 μ A is reached. The chosen etching current produces sharp tips at the liquid surface. The etched sample immediately afterwards is subjected to ultrasonic cleaning by using an ultrasonic device. The cleaned tip is then mounted in a standard, home-built, field emission microscope (FEM) with a tip-screen distance of 10 mm [10]. The anode is formed as a phosphored tin-oxide coated conductive glass screen in order to allow for the recording of the emission current and the electronic images [9].

The FEM was evacuated to ultra high vacuum (UHV) conditions using a mechanical rotary pump that produces pressures of about 10^{-3} mbar and an oil diffusion pump system, connected to a liquid nitrogen (LN_2) trap which leads to finally

reaching a base pressure of about 10^{-9} mbar [11, 12]. Then, the samples are subjected to sample conditioning treatment which consists of an initial baking of the system for 12 hours at 170 °C, a follow-up baking of the system for 12 hours, thermal relaxation for 12 hours at 170 °C, and finally cooling the sample by liquid nitrogen – while observing the emission behavior. This allowed recording the effects of these conditioning processes on carbon fiber tips.

To record the emission behavior, an additional high tension (EHT) power supply is used to apply the voltage on the tip. This is slowly increased until the emission current rises to about one microampere and is measured on a Keithley 485 autoranging picoammeter. The applied voltage is then slowly decreased until the emission current vanishes. Within this range, a linear Fowler-Nordheim (FN) plot is expected [13].

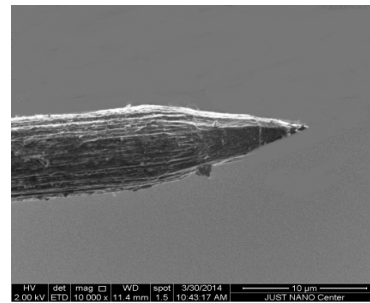


FIG.1. Scanning electron micrograph of a very sharp carbon fiber tip at 10000 \times magnification.

Results and Discussion

The emission characteristics derived will be presented as the I-V characteristics and the corresponding Fowler-Nordheim (FN) plots whose equation was given in the introduction. The slope obtained from these plots enables calculating the tip apex radii. These radii of the carbon fiber tips have been measured as the average of the graphically best-fitting circles of the SEM employed in the experiments.

The latter ones are used to interpret the experimental data and extract relevant information such as the apex radii, which is an important data needed for further physical analysis in a future research. During the experiments, electronic emission images have been recorded by a standard digital camera to study the spatial distribution and stability of the emission current. Stability as well as brightness (emitted current/unit emitting area/unit solid angle where the electrons are emitted out of the

surface) are important factors for judging the quality of the electron source for practical applications.

During the sample conditioning treatment, it was discovered that there were statistical variations in the electronic emission of the

various tip microemitters under the corresponding UHV conditions [14].

Very Sharp Tips

In this part, one of the very sharp carbon fiber tips (such as the one shown in Fig. 1) has been tested during sample conditioning treatment. The apex radius of this tip was 57 nm.

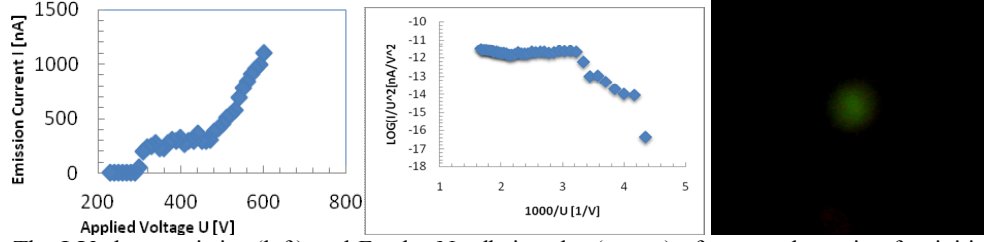


FIG. 2. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a very sharp tip after initial baking for 12 hours at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber very sharp tip after initial baking for 12 hours at 1 μA emission current. The slope of the linear part of the plot is $S = 1034$.

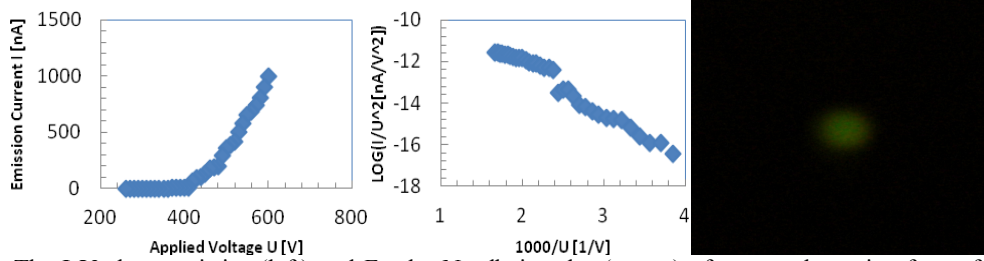


FIG. 3. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a very sharp tip after a follow-up baking for 12 hours at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber very sharp tip after a follow up baking for 12 hours at 1 μA emission current. The slope is $S = 2407$.

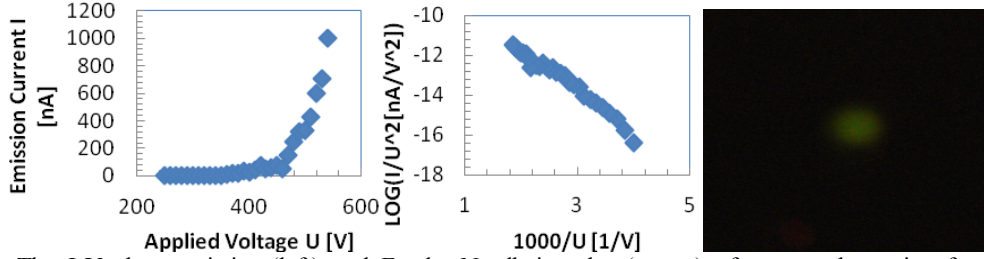


FIG. 4. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a very sharp tip after thermal relaxation process for 12 hours. The emission image (right) is obtained from a carbon fiber very sharp tip after thermal relaxation process overnight at 1 μA . The slope is $S = 2027$.

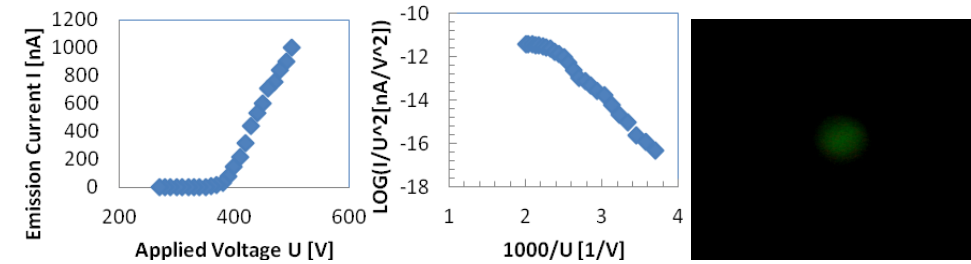


FIG. 5. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a very sharp tip during cooling process. The emission image (right) is obtained from a carbon fiber very sharp tip during cooling process at 1 μA emission current. The slope of the FN plot is $S = 3069$.

After initial baking, as shown in Fig. 2, the F-N plot shows a non-linear behavior, and the emission current was unstable. The follow-up baking, as shown in Fig. 3, shows a disconnected plot because the voltage drops from 66 V to 5.5 V, possibly due to a change in emitter shape. After the sample conditioning, the emission current stability becomes much higher as shown in Figs 4 and 5. The emission images as shown

in Figs 2 - 5 become brighter, more stable and more focused.

Sharp Tips

In the second part, we tested one of the sharp carbon fiber tips which has been tested during sample conditioning treatment. The apex radius of this tip was 70 nm.

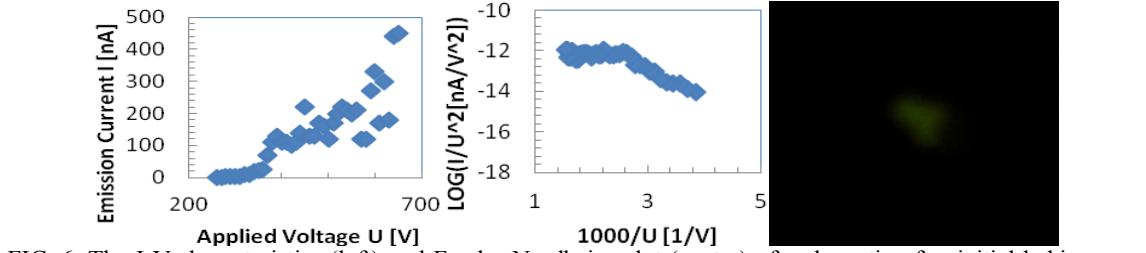


FIG. 6. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a sharp tip after initial baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber sharp tip after initial baking for 12 hours at 1 μA . The slope is $S = 753$.

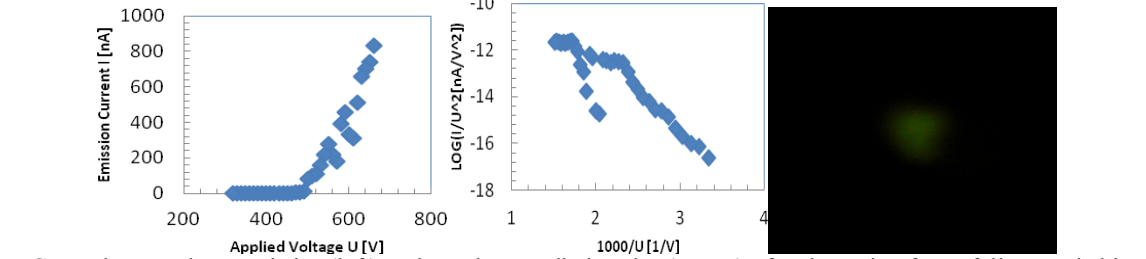


FIG. 7. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a sharp tip after a follow-up baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber sharp tip after a follow-up baking for 12 hours at 1 μA . The slope is $S = 2577$.

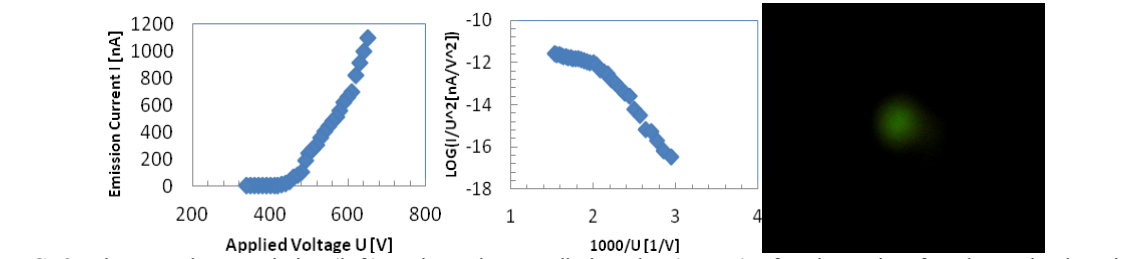


FIG. 8. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a sharp tip after thermal relaxation process over night. The emission image (right) is obtained from a carbon fiber sharp tip after thermal relaxation process treatment for 12 hours at 1 μA . The slope is $S = 2472$.

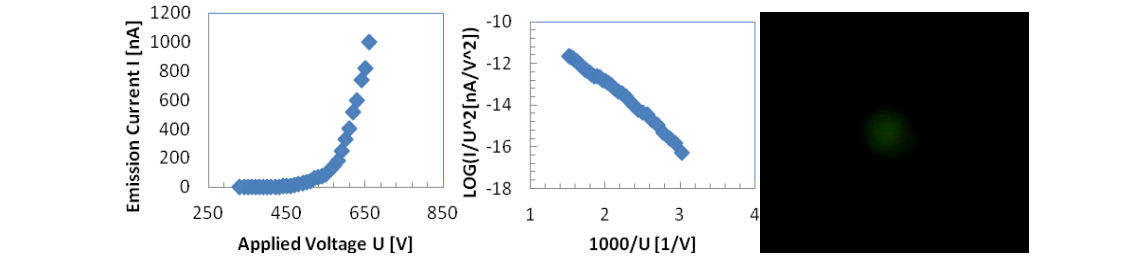


FIG. 9. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a sharp tip during cooling process. The emission image (right) is obtained from a carbon fiber sharp tip during cooling process at 1 μA . The slope is $S = 2897$.

After the baking treatment, the emission behavior showed little variation on the tip between the initial baking and the follow-up baking as shown in Figs 6 and 7. The influence of the thermal relaxation and cooling the sample to the LN₂ temperature on the emission characteristics was studied. There was significant variation in the magnitude of the emission current and its stability as shown in Figs 8 and 9. Figs 6 - 9 show an improvement

in the emission images during these treatments [14].

Medium Sharp Tips

In the third investigation, one of the medium sharp carbon fiber tips has been tested during sample conditioning treatment. The apex radius of this tip was 90 nm.

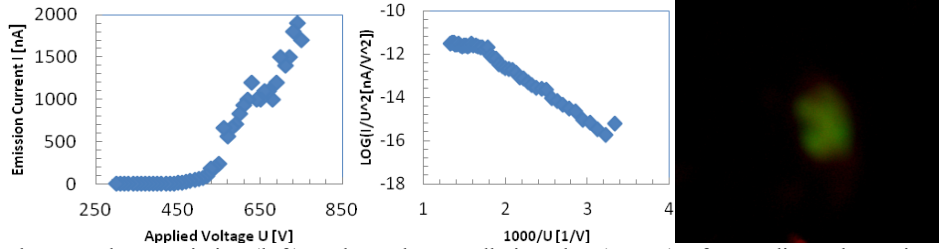


FIG. 10. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a medium sharp tip after initial baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber medium sharp tip after initial baking for 12 hours at 1 μA . The slope is $S = 2274$.

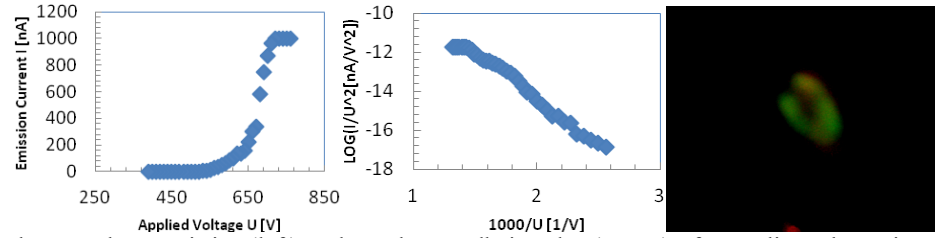


FIG. 11. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a medium sharp tip after second baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber medium sharp tip after a follow-up baking for 12 hours at 1.2 μA . The slope is $S = 4574$.

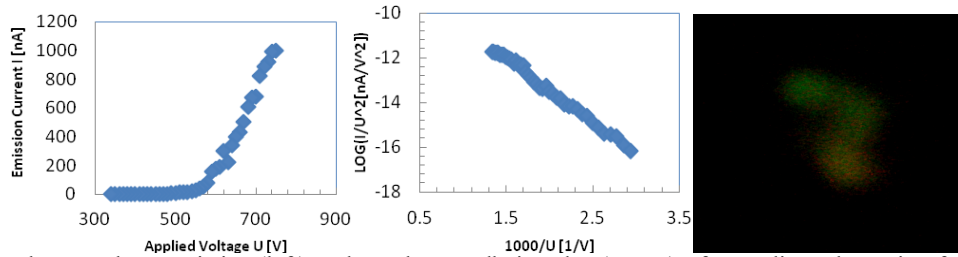


FIG. 12. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a medium sharp tip after thermal relaxation process over night. The emission image (right) is obtained from a carbon fiber medium sharp tip after thermal relaxation process for 12 hours at 1 μA emission current. The slope of the FN plot is $S = 2856$.

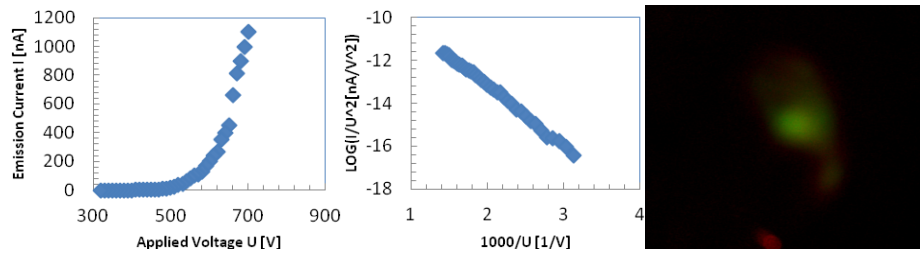


FIG. 13. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a medium sharp tip during cooling process. The emission image (right) is obtained from a carbon fiber medium sharp tip during the cooling process (down to LN₂ temperature) at 1.2 μA . The slope is $S = 2824$.

The field electron emission characteristics from the follow-up baking did not show much variation from those characteristics of the initial baking as shown in Figs 10 and 11. The last two processes, thermal relaxation and cooling process, cause a clear improvement in the emission current stability as shown in Figs 12 and 13 [10, 15].

Blunt Tips

In the fourth study, one of the blunt carbon fiber tips has been tested during sample conditioning treatment where the apex radius of this tip was 150 nm.

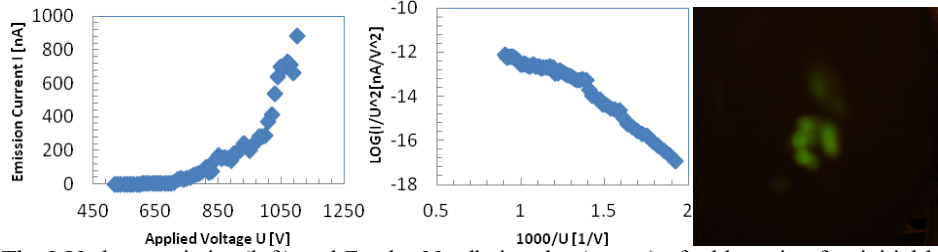


FIG. 14. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a blunt tip after initial baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber blunt tip after initial baking for 12 hours at 1 μA . The slope is $S = 4508$.

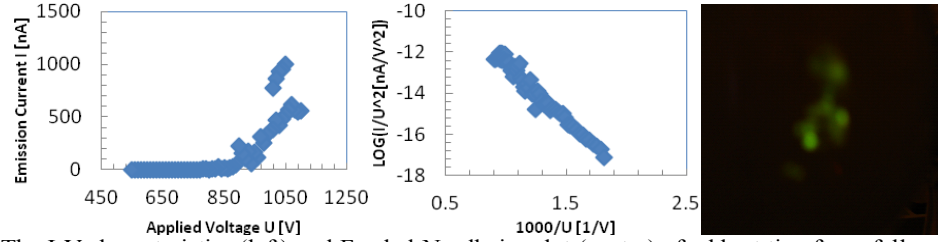


FIG. 15. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a blunt tip after a follow-up baking over night at a temperature of 170°C. The emission image (right) is obtained from a carbon fiber blunt tip after a follow-up baking for 12 hours at 1 μA . The slope is $S = 5558$.

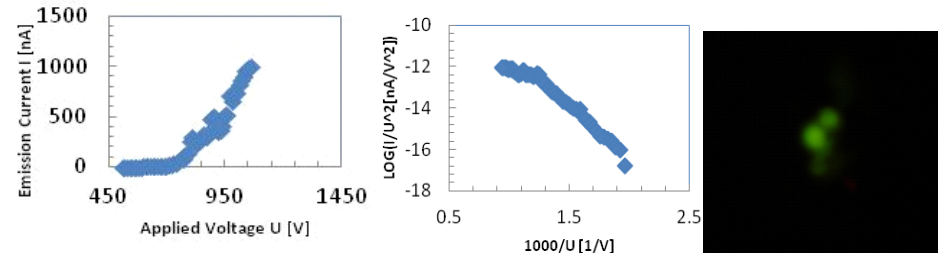


FIG. 16. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a blunt tip after thermal relaxation over night. The emission image (right) is obtained from a carbon fiber blunt tip after thermal relaxation for 12 hours at 1 μA . The slope is $S = 4196$.

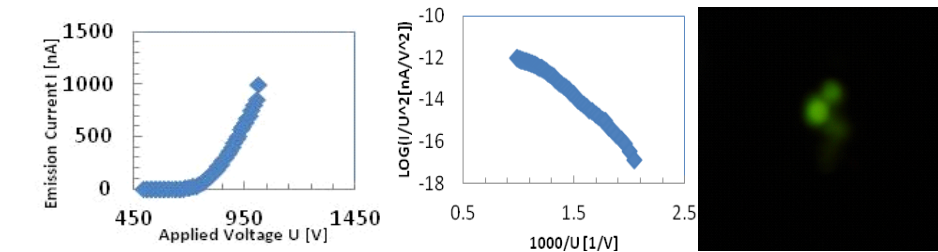


FIG. 17. The I-V characteristics (left) and Fowler-Nordheim plot (center) of a blunt tip during cooling process. The emission image (right) is obtained from a carbon fiber blunt during cooling process at 1 μA . The slope is $S = 4361$.

Following this sample conditioning, as shown in Figs 14 – 17, the typical I - V characteristics exhibited a reduction in the hysteresis effect and the emission current became more stable. Lower field produced the same values of current, and the I-V characteristics produced a linear F-N plot [16]. The electron emission images have shown to possess more stability and higher brightness. Principally, this is comparable with Yahachi Saito and Uemura work while investigating applications to electron sources [17]. Carbon fibers could be further investigated for applications where they were used to fabricate nanometer sized tips at their ends [18-20] and as a tip-based electron source [21]. The results reported here could enrich the theoretical investigations to understand the physical analysis of the reported phenomena. This, in addition to the mechanism assumed to control the emission of electrons from the fibers studied, will be the subject of further investigation

Conclusions and Future Work

Carbon fiber emitters were prepared using an electrolytically etching process. These emitters have been tested within the FEM system. During the conducted experiments, the current values were measured with a change in the voltage values. These experiments were carried out using carbon fibers with different apex radii. The analytical facility used enabled measuring various characteristics of the field electron emission. The sample conditioning procedures that included initial baking, a follow-up baking, thermal relaxation and cooling process down to LN₂ temperatures of about (-196°C), produced an improved stability in the emission current and higher brightness. Thus, the performance of these tips was found to be highly dependant on the utilized sample conditioning treatment. Future work will involve the application of this research findings within controlled-switching.

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