

# Enhanced Field Electron Emission from Dielectric Coated Highly Emissive Carbon Fibers

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This paper describes experiments aimed at characterizing the behavior of field electron emitters fabricated by coating carbon fibers with epoxy resin. Polyacrylonitrile carbon fibers of type VPR-19, thermally treated at 2,800°C, were used. Each was initially prepared in a “uncoated” state, by standard electro polishing and cleaning techniques, and was then examined in a scanning electron microscope. The fiber was then baked overnight in a field electron microscope (FEM) vacuum chamber. Current-voltage characteristics and FEM images were recorded on the following day or later. The fiber was then removed from the FEM, coated with resin, “cured” by baking, and replaced in the FEM. After another overnight bake, the FEM characterization measurements were repeated. The coated fibers had significantly better performance than uncoated fibers. This confirms the results of earlier experiments, and is thought to be due in part to the formation of a conducting channel in the resin over layer. For the coated fiber, lower voltages were needed to obtain the same emission current. The coated fibers have current-voltage characteristics that show smoother trends, with greater stability and repeatability. No switch-on phenomena were observed. In addition, the emission images on the phosphor-coated FEM screen were more concentrated, and hence brighter.

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## INTRODUCTION

In 1879, Thomas Edison fabricated carbon fibers by baking cotton threads or bamboo slivers at high temperatures carbonizing them into an all-carbon fiber filament used in one of the first incandescent light bulbs to be heated by electricity, in 1879 in 1880, Lewis Latimer developed (American Chemical Society, 2014). Carbon fibers have many properties that are desirable for field electron emission, such as low density, high thermal stability, and chemical stability in the absence of oxidizing agents, good thermal and electrical conductivity, and excellent creep resistance (Baker et al., 1974; Huang, 2009).

Polyacrylonitrile (PAN) carbon fibers polymerized from acrylonitrile and containing 68% carbon were developed for industrial applications, including cathodes for electron

sources (Morita et al., 1986). Properties making PAN fibers useful as electron emitters include the stability of the fiber surface under ion bombardment and resulting long lifetime (Mousa, 1996). For cold field electron emission (CFE) from uncoated or coated carbon fibers into vacuum, a local electrostatic field (at the emitter surface) of more than 3 V/nm is required (Forbes, 2008). In CFE, the applied electric field reduces the surface potential barrier, enabling electrons at the Fermi level to tunnel through this barrier (Forbes, 2004; Fowler & Nordheim, 1928). Carbon fibers can emit stable currents for long periods of time under poor vacuum conditions, with greater resistance to ion bombardment than metal emitters possess. Coating carbon fibers with dielectric materials produces an increase in the emission current, improves its stability, and increases the emitter lifetime (Mousa & Kelly, 2003). This paper describes some new CFE

experiments with coated carbon fibers. The ultimate aim is to obtain improved performance, including generation of emission currents at lower applied voltages.

## MATERIALS AND METHODS

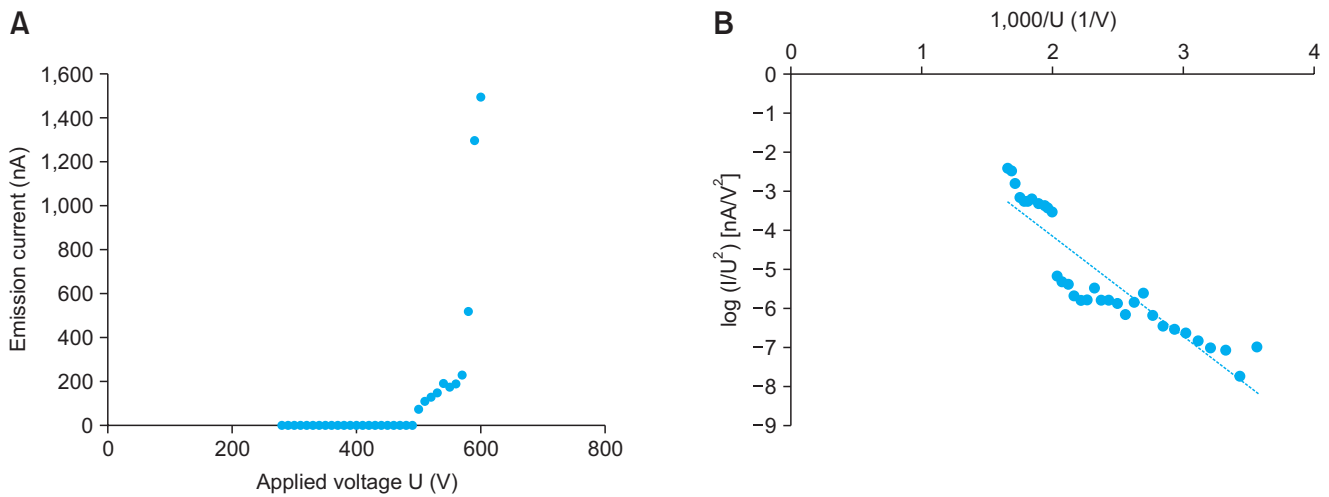
The micropoint emitters used for these measurements were electrolytically etched from 7  $\mu\text{m}$  diameter carbon fibers using a 0.1 M solution of NaOH. Typically, etching requires a current of 35  $\mu\text{A}$  at a voltage of 2 V. This is followed by ultrasonic cleaning to remove the etching residues.

The etched fibers are first examined in a scanning electron microscope, which allows measurements to be made to

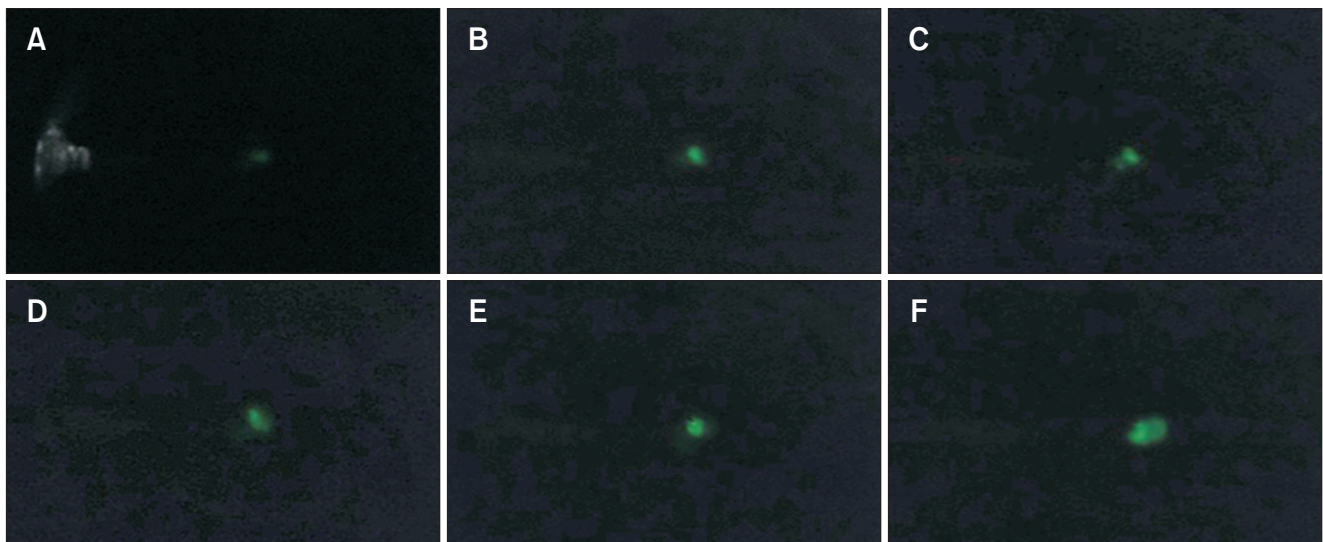
determine the apex radius of the tip of the emitter. These tip-radius results, together with estimates made from field electron microscope (FEM) experiments described below, have already been reported elsewhere (Forbes et al., 2016).

The etched carbon fibers were then placed in the FEM vacuum chamber and baked for 12 hours at 180°C. With the aid of a liquid nitrogen (LN2) trap in the pumping path, it was possible to obtain ultra-high vacuum, at a pressure-level of around  $10^{-8}$  mbar.

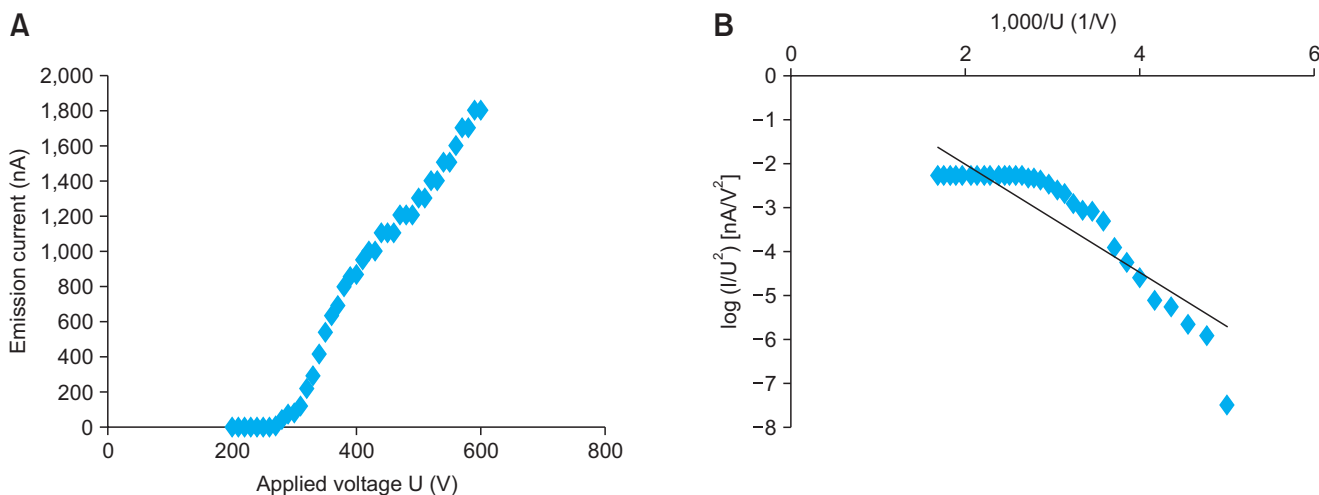
Characterization experiments were then performed in the FEM, as follows. For each fiber: (a) the current-voltage ( $I$ - $V$ ) characteristics were measured, to allow a Fowler-Nordheim plot (FN plot) to be drawn; (b) FEM images (formed when



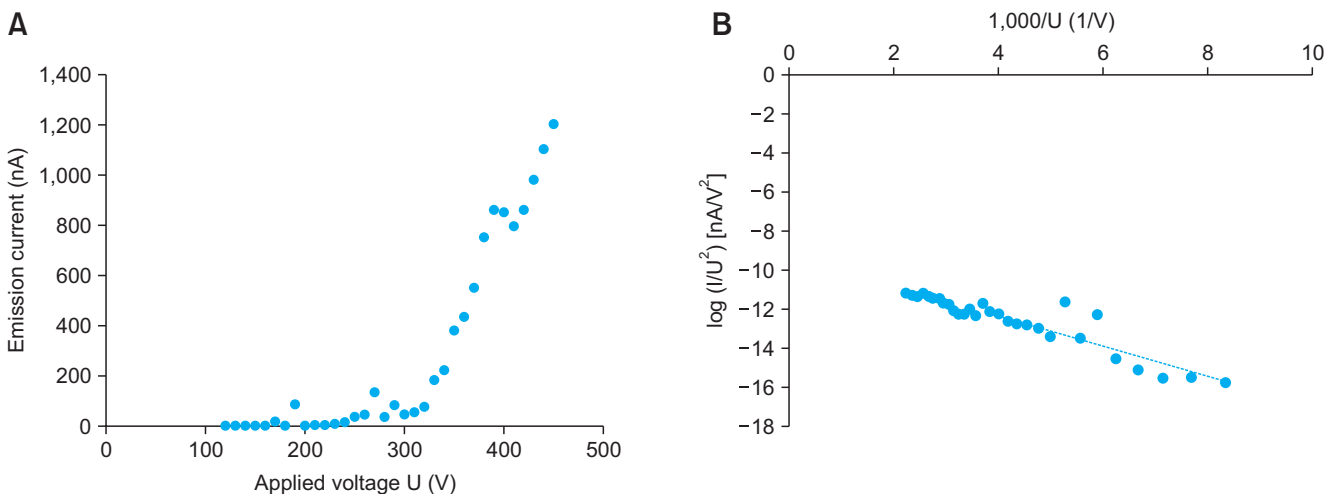
**Fig. 1.** (A) The  $I$ - $V$  characteristics when increasing the voltage for an uncoated carbon fiber tip; (B) the corresponding Fowler-Nordheim plot, where the slope of the fitted line (determined by least-squares regression) is  $-2,570$  decade volt.



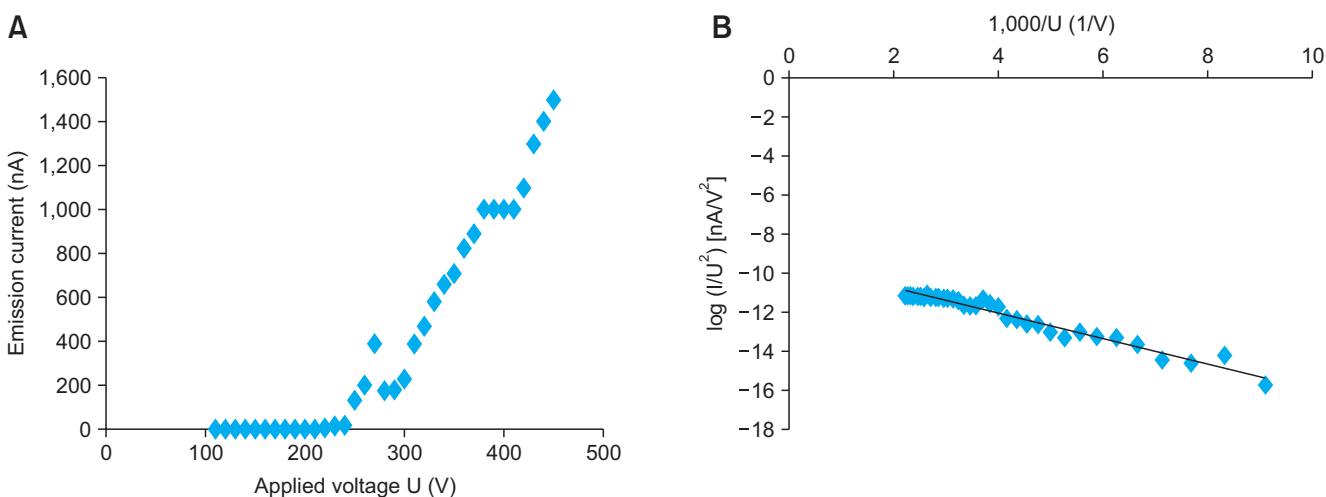
**Fig. 2.** Field electron microscope images of cold field electron emission from an uncoated carbon fiber tip. As the applied voltage is increased, the image becomes brighter and larger. These images were taken under the following conditions: (A) 540 V, 0.19  $\mu\text{A}$ ; (B) 550 V, 0.18  $\mu\text{A}$ ; (C) 560 V, 0.19  $\mu\text{A}$ ; (D) 570 V, 0.27  $\mu\text{A}$ ; (E) 580 V, 0.52  $\mu\text{A}$ ; (F) 590 V, 1.3  $\mu\text{A}$ .



**Fig. 3.** (A) The *I-V* characteristics of a carbon fiber tip before coating, for decreasing voltage; (B) corresponding Fowler-Nordheim plot, where the slope of the trend line is  $-1,220$  decade volt.



**Fig. 4.** (A) The *I-V* characteristics of cold field electron emission from a coated carbon fiber tip, for increasing voltage; (B) the corresponding Fowler-Nordheim plot, where the trend-line slope is  $-760$  decade volt.



**Fig. 5.** (A) The *I-V* characteristics of a coated carbon fiber, for decreasing voltage; (B) corresponding Fowler-Nordheim plot.

the emitted current hits the phosphor-covered viewing screen that acts as the anode) were recorded; and (c) the stability of the emitted current was assessed (Forbes et al., 2015; Latham & Mousa, 1986; Madanat et al., 2015). The distance between the carbon fiber emitter and the screen anode was 10 mm.

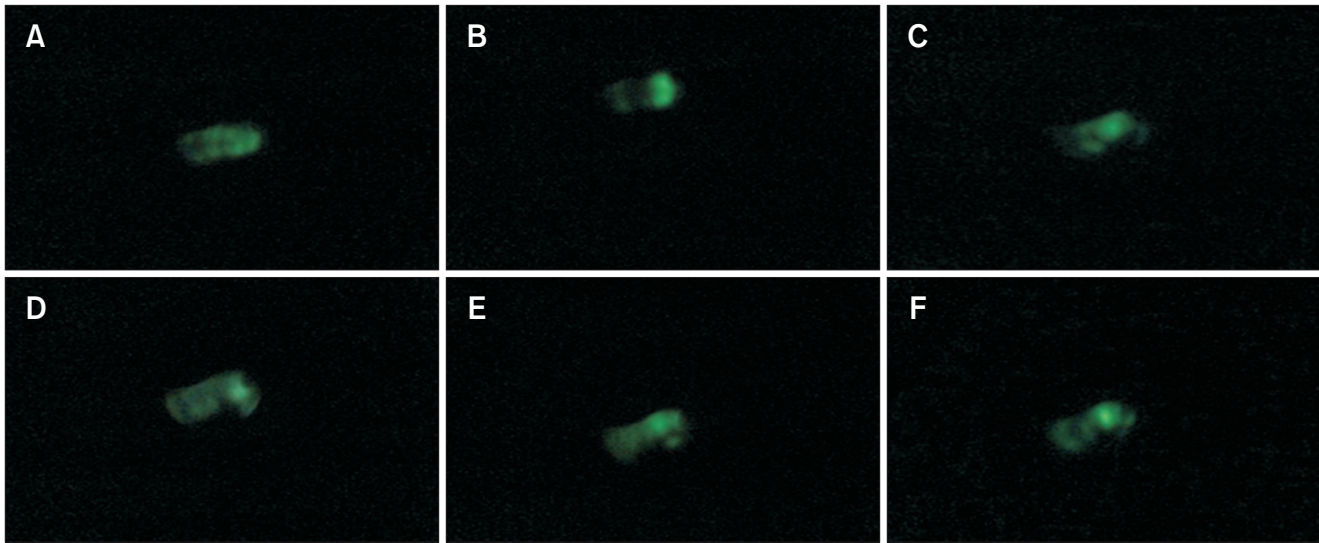
After an uncoated carbon fiber had been examined as described above, it was removed from the FEM and coated with dielectric epoxy resin. Each carbon fiber was slowly dipped into the resin (Clark Electromedical Instruments Corporate, USA) twelve times, with each dip forming a layer of about 15 nm. Hence, the final layer thickness was about 150 to 200 nm. This was followed by baking the fiber emitter in an oven for 0.5 hour at 100°C, to remove the solvents, followed by 0.5 hour at 190°C to cure the resin, as described by Mousa & Kelly (2003).

Then, the coated emitters were examined in a FEM, in the same way as described above for the uncoated emitters.

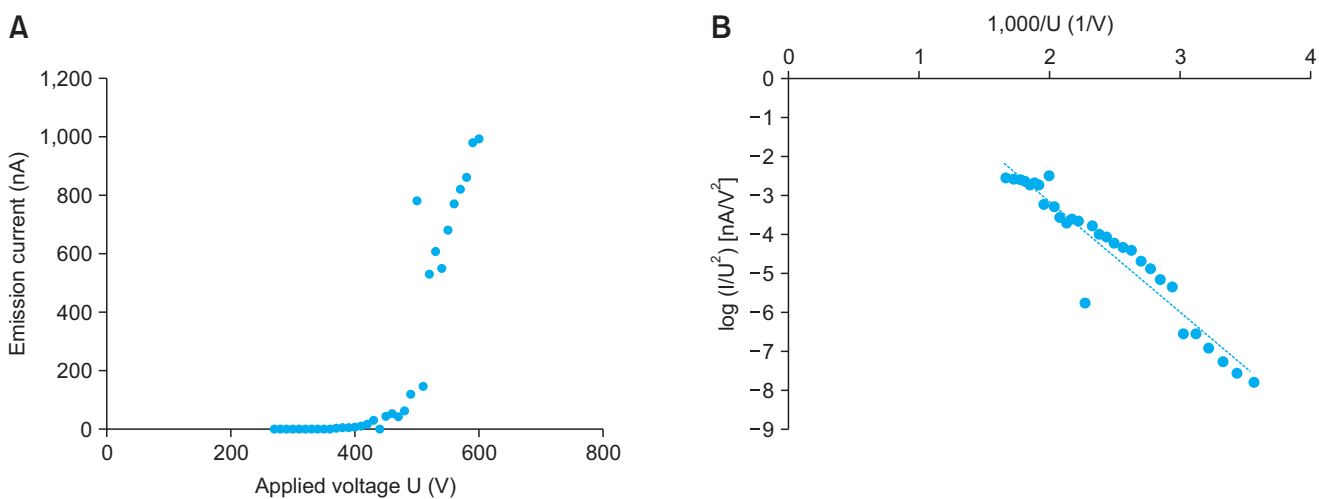
## RESULTS

In the FEM experiments, a series ballast resistor of 1 MΩ was used to protect the emitter, as described long ago by Braun et al. (1975). In Fig. 1A the  $I$ - $V$  characteristics are recorded for an uncoated carbon fiber tip that shows a sharp turn-on as voltage increases. Fig. 1B shows the corresponding FN plot; this has a slope of  $-2,570$  decade volts.

In what follows, the unit “decade volt” is abbreviated as “dec V”. On the vertical axes of the FN plots, the symbol “log” means “ $\log_{10}$ ”; for clarity in related calculations, logarithms to base 10



**Fig. 6.** Field electron microscope images recorded for coated carbon fiber, for increasing voltage: (A) 380 V, 0.75  $\mu$ A; (B) 390 V, 0.86  $\mu$ A; (C) 400 V, 0.90  $\mu$ A; (D) 420 V, 0.92  $\mu$ A; (E) 440 V, 1.1  $\mu$ A; (F) 450 V, 1.2  $\mu$ A.



**Fig. 7.** (A)  $I$ - $V$  characteristics of an uncoated carbon fiber tip, for increasing voltage; (B) corresponding Fowler-Nordheim plot, where the slope of the trend line is  $-2,820$  decade volt.

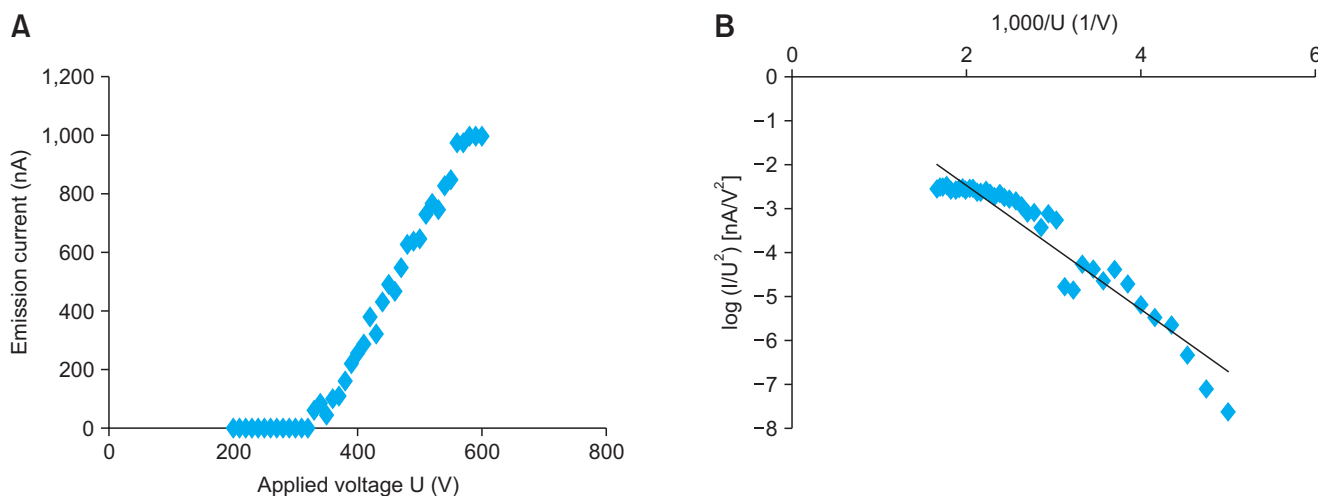
are considered in field emission contexts to be measured in “decades”.

Fig. 2 shows some fluctuations in the electron emission image where the current may have decreased as the voltage was increased. This shows the instability in the electron emission with an uncoated carbon fiber, as reported by Braun et al. (1975). Fig. 3A shows the smooth *I-V* characteristics for an uncoated tip as the voltage is decreased; Fig. 3B shows some of the points around the trend line for the corresponding FN plot, which has a mean slope of  $-1,220$  dec V.

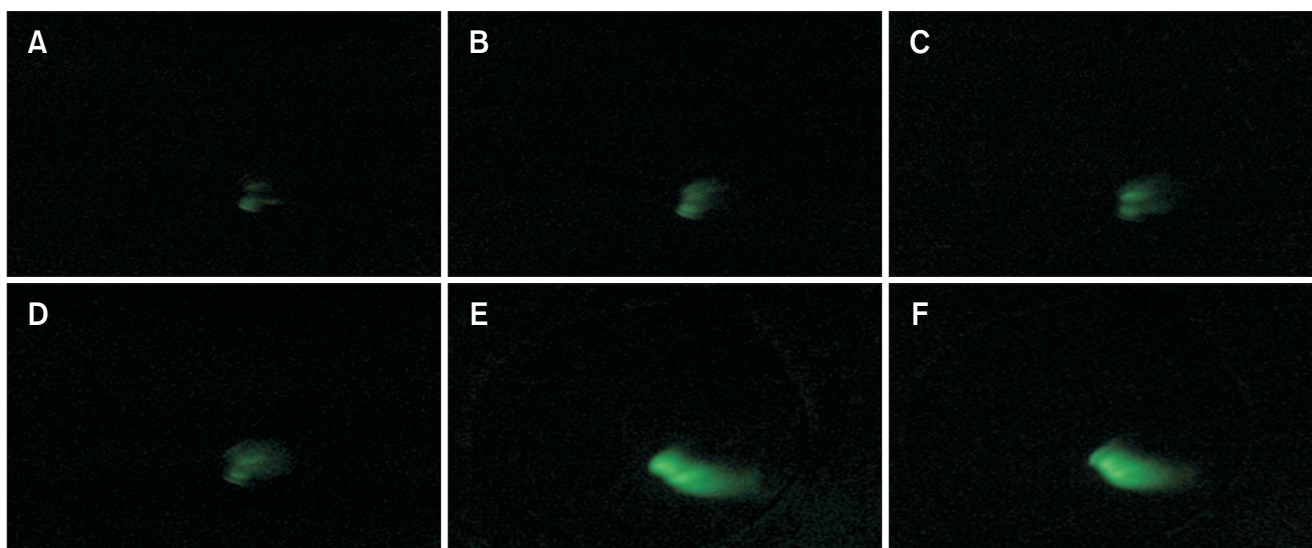
Fig. 4A shows current-voltage characteristics for the carbon fiber after coating. This plot (which is for voltage increasing) is smoother than was seen with the uncoated carbon fiber. Electron emission begins at a lower voltage and there is

much less fluctuation in the electron emission, both when the voltage is increasing and when it is decreasing. The FN plot slope in Fig. 4B (for a coated carbon fiber, for increasing voltage) is much less in magnitude than the slope for the uncoated carbon fiber. In 1986, Latham and Mousa showed a similar reduction in slope magnitude when the same dielectric was used to coat a tungsten emitter.

Fig. 5 shows the current-voltage characteristics and the corresponding FN plot for a coated carbon fiber, for decreasing voltage. The FN plot slope is  $-650$  dec V. In Fig. 4 and 5 (relating to the coated carbon fibers), the individual data points on the FN plot are much closer to the trend line than in Fig. 1 and 3 (for the uncoated fibers). The least scatter in the FN plot is in Fig. 5 (which is for the coated fiber, for



**Fig. 8.** (A) *I-V* characteristics of an uncoated carbon fiber tip, for decreasing voltage; (B) corresponding Fowler-Nordheim plot, where the slope of the trend line is  $-1,410$  decade volt.



**Fig. 9.** Field electron microscope images of cold field electron emission from an uncoated carbon tip, for increasing applied voltage. These images were taken at: (A) 550 V, 0.68  $\mu$ A; (B) 560 V, 0.77  $\mu$ A; (C) 570 V, 0.82  $\mu$ A; (D) 580 V, 0.98  $\mu$ A; (E) 590 V, 0.98  $\mu$ A; (F) 600 V, 1  $\mu$ A.

decreasing voltage).

A second carbon fiber sample was studied, using the procedures described. Some fluctuations were observed in the FEM image shown in Fig. 6. Fig. 7, 8, and 9 present  $I$ - $V$  characteristics and FN plots for this second sample. These correspond to the results shown for the first sample in Fig. 1, 3, 4, and 5.

The  $I$ - $V$  characteristics for decreasing voltage, as shown in Fig. 10, show smoother behavior in the FN plot, with fewer irregular points around the trend line. Some fluctuations were observed in the FEM image shown in Fig. 11.

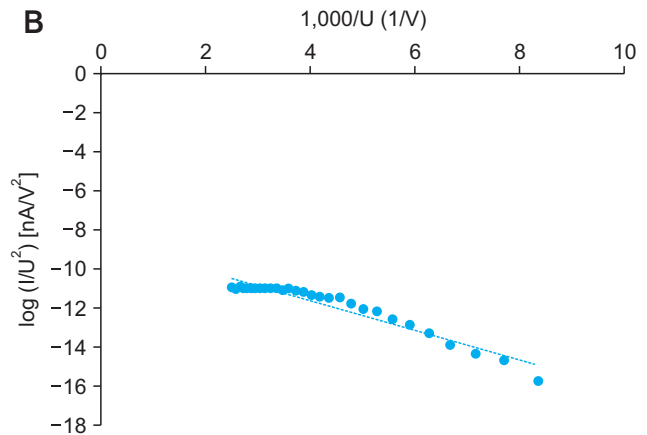
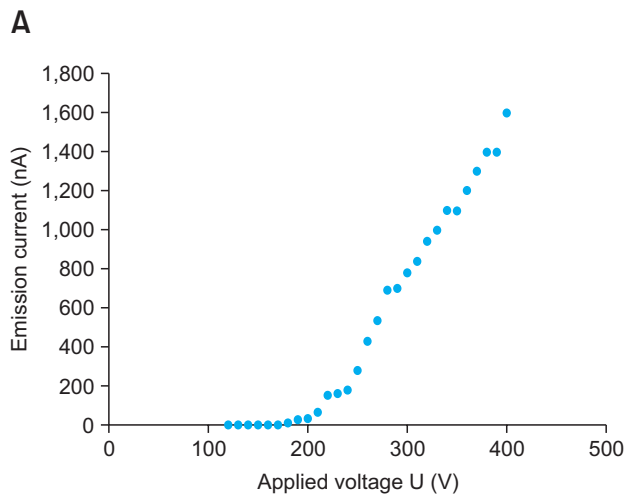
The measurements for this coated carbon fiber also exhibit smooth behavior of the  $I$ - $V$  characteristics, in both the increasing and decreasing cycles, with the data points for the FN plot being more regular for both cycles. The plot has a

slope that is lower in magnitude for the coated fiber than for the uncoated fiber.

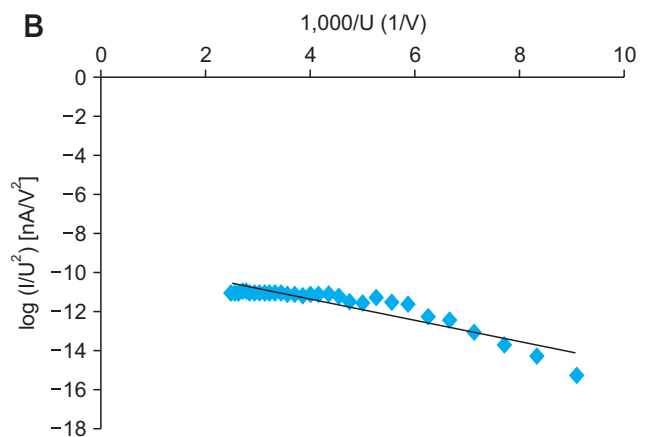
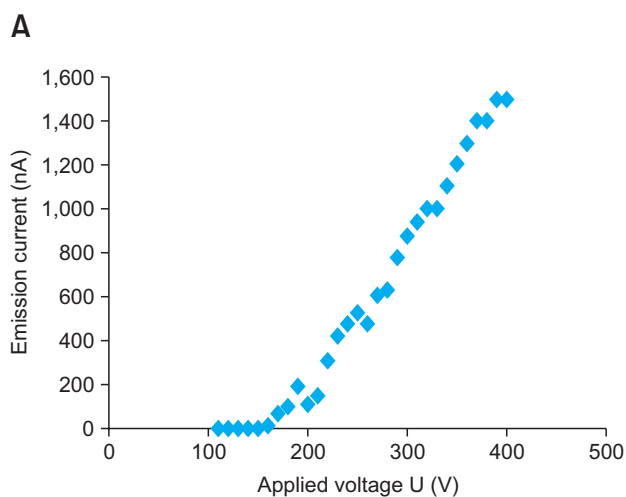
The FEM images in Fig. 12, which were recorded at several voltages, show that the emission area is small and the current is stable.

## DISCUSSION AND CONCLUSIONS

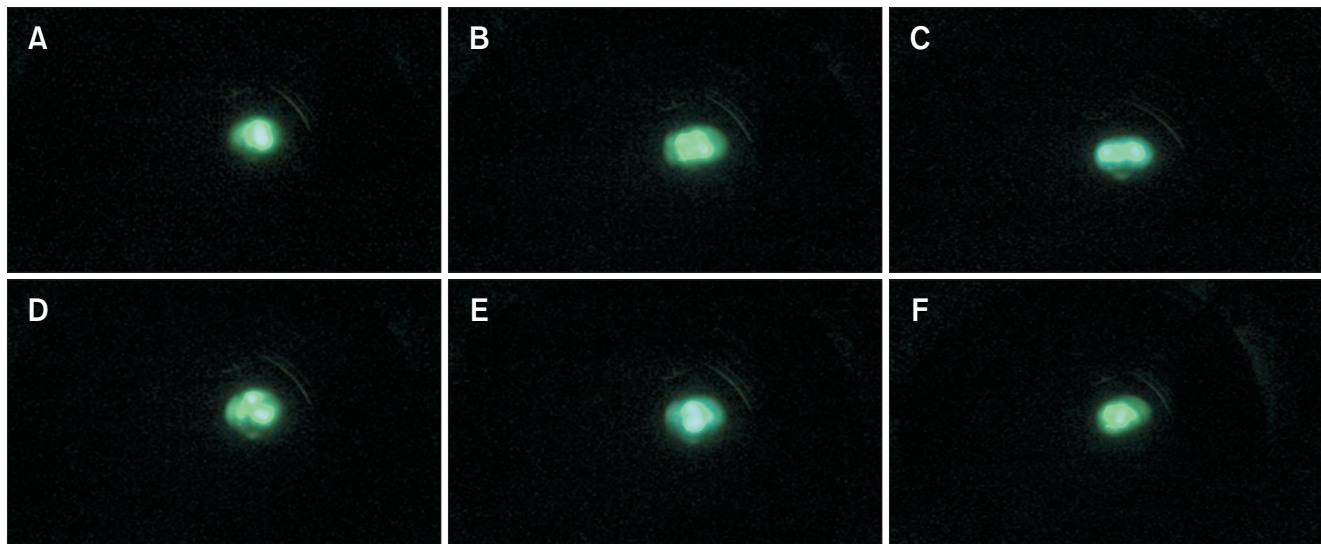
The effects of the epoxy resin coating may be described as follows. Good results were obtained with the coated emitters, with emission currents of a few microamperes obtained at voltages as low as 460 V. These voltages were significantly lower than those needed to obtain the same emission current with uncoated tips. Further, the coated tips have  $I$ - $V$  characteristics that show smoother trends, and greater



**Fig. 10.** (A)  $I$ - $V$  characteristics of the second coated carbon fiber tip, for increasing voltage; (B) corresponding Fowler-Nordheim plot, where the trend-line slope is  $-760$  decade volt.



**Fig. 11.** (A)  $I$ - $V$  characteristics of the second coated carbon fiber tip, for decreasing voltage; (B) corresponding Fowler-Nordheim plot, where the trend-line slope is  $-541$  decade volt.



**Fig. 12.** Field electron microscope images recorded for the second coated carbon fiber tip, for increasing voltage: (A) 300 V, 780 nA; (B) 310 V, 848 nA; (C) 320 V, 940 nA; (D) 340 V, 1  $\mu$ A; (E) 350 V, 1  $\mu$ A; (F) 350 V, 1.1  $\mu$ A.

**Table 1.** Current-voltage characteristics of carbon fiber emitters before and after coating with epoxy resin

Tip	Clean-tip characteristics		Composite -tip characteristics	
	Applied voltage range (V)	Emission current range (nA)	Applied voltage range (V)	Emission current range (nA)
Sample number 1				
Increasing voltage	280-600	0.0086-1,500	120-450	0.0024-1200
Decreasing voltage	600-200	1,600-0.0013	450-110	1,500-0.0022
Sample number 2				
Increasing voltage	280-600	0.0013-160	120-400	0.0026-1,600
Decreasing voltage	600-200	1,600-0.001	400-110	1,500-0.0065

stability and repeatability than before coating. In contrast with the effects observed with dielectric-coated metallic emitters (Mousa, 2007), no switch-on phenomena were observed, and the effects of thermal processing and relaxation procedures did not change the stability. In addition, the emission images were more concentrated on the phosphor-coated FEM screen, and correspondingly brighter.

Table 1 shows the effects of the epoxy resin coating on the emission characteristics of the carbon fiber emitters. Of particular note is the remarkable improvement in emission current values after coating. It is thought that this is due to the formation of a conducting channel in the dielectric layer on the tip apex, and that the field at the top (vacuum-end) of this channel is greater than the apex field of the same emitter before coating. This is a form of field enhancement effect, and consequently the fiber needs lower applied voltage in order to emit a given value of emission current.

In summary, coating carbon fibers of the VPR-19 PAN type with dielectric epoxy resin improves the electron emission properties in various ways. These include: lowering the required voltage for a given emission current, compressing the electron image (thereby making it brighter for a given emission current), and improving the stability of the current-

voltage characteristics. In general, the magnitude of the FN plot slope is reduced after applying the coating.

These confirmed improvements in the performance of our composite electron sources, as compared with uncoated emitters, are of significant technological interest in the context of building small bright electron sources that are robust in industrial-vacuum conditions. We intend to extend this work by investigating the effects of other forms of coating.

## CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

## ACKNOWLEDGMENTS

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