

# Experimental Characterization of a Carbon Nanotube Field Emission Cathode

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There is interest in the use of carbon nanotubes (CNTs) to create a field emission cathode. The primary benefit of this cathode is that a gas flow is not required to create electrons, which is of critical importance for space missions that must minimize propellant mass. A field emission cathode consisting of CNT arrays is fabricated by the Georgia Tech Research Institute and the Georgia Tech High-Power Electric Propulsion Laboratory. The cathode is tested at pressures below  $3 \times 10^{-5}$  Torr. The average output current density is measured over a cathode voltage range of 250-800 V relative to the gate for several CNT cathodes. The highest stable emission current density is 0.6 mA/cm<sup>2</sup>. The emission current density is measured throughout a 50-hour life test at a cathode voltage of 550 V which demonstrated an overall constant emission current density of 0.51 mA/cm<sup>2</sup> with an emission current to input power ratio of 1.7 mA/W.

## I. Introduction

THE use of carbon nanotubes (CNTs) for field emission (FE) cathodes is of interest for use as a general electron emitter, especially in the area of electric propulsion (EP). There is a need for an efficient electron source to neutralize the exhaust plume in low-power EP devices.<sup>1</sup> Most thrusters operate in conjunction with thermionic hollow cathodes, which require a gas flow in order to emit electrons. For a hollow cathode in use with a 200-W Hall effect thruster (HET) the propellant flow rate through the cathode is typically an additional 10% of the flow through the thruster itself. In addition, hollow cathodes are relatively complex in that they require a heater element, which is an additional load on the spacecraft power system. Since most low-power EP systems have limited power capacity, any expenditure in power that does not directly generate thrust is a source of inefficiency. Rather than use a hollow cathode, it is suggested that CNT cathodes be used for plume neutralization. Recent work demonstrates the potential of FE cathodes not only in the areas of low power EP, but also in the areas of propellant-less propulsion such as space tethers, and spacecraft charge control.<sup>1,2</sup>

There are numerous advantages to using CNT cathodes. Unlike hollow cathodes, CNT cathodes require no gas flow to operate, and require no additional components such as a heater element. This simplicity reduces extraneous loads on the spacecraft systems and allows for greater design flexibility. There are also numerous benefits to CNT cathodes derived from the nature of the cathode's construction. Electron emission does not originate from any one point, but rather is spread across many single nanotubes, namely those where the local electric field from the cathode gate to the CNTs exceeds a minimum value required to extract electrons, called the turn-on value. For the cathodes tested, the average turn-on electric field is 1.57 V/ $\mu\text{m}$ . As long as the nanotubes all have approximately the same height, and thus approximately the same separation distance from the end of the nanotubes to the gate screen, the electric field is evenly distributed across multiple nanotubes. Unlike thermionic emission, high temperature is not required to lower the work function for emission, which makes CNT cathodes very durable, both mechanically and thermally. Prior work demonstrates the durability of field emission cathodes compared to thermionic emission cathodes.<sup>3</sup> Another benefit to CNT cathodes is that they can be easily scaled to provide any current determined by

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the constructed configuration. CNT cathodes are fabricated as thin, lightweight films and as such can be applied onto the surface of the thruster body itself rather than a bulky tube offset from the thruster. The size of the emitter can be increased simply by adding additional CNTs with only a negligible increase in mass and complexity. This situation also illustrates the inherent redundancy in CNT cathodes, as they consist of many electron sources in parallel. Thus CNT cathodes have great potential as a general purpose, propellant-less electron source.

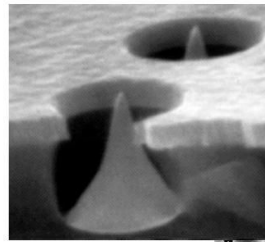
C. A. Spindt developed the first field emission cathode in the late 1960's, termed the Spindt cathode. A Spindt cathode consists of two conducting sheets separated by an insulating layer. Figure 1 shows, a series of holes are cut into the top conducting layer and the insulating layer, with metal cones attached to the bottom conductor. This top conductor is termed the gate, with the lower conductor the base. To operate a Spindt cathode, a bias is placed across the gate. This bias is positive relative to the base and when it is large enough field emission will occur. The bias required to initiate field emission has an average value of about 100 V. Past operations of Spindt array cathodes show that Spindt cathode emission is approximately 100 mA/cm<sup>2</sup> at about 7 mA/W.<sup>4</sup> The advantages to a Spindt cathode came from its propellant-less nature, as well as a low mass, low power consumption, and clean operation.<sup>5</sup> However, Spindt cathodes suffer from several limitations. First, the emission current from a Spindt cathode is unstable, as the cones are prone to form surface oxides which increase the work function and thus the operating voltage. Another drawback to Spindt cathodes is due to sputtering from any ionized propellant. As the tip sputters, the radius of the tip increases due to the tip geometry (termed a "sharpened pyramid"). As the radius of the tip increases, the concentration of electric field lines converging on any one location on the tip decreases. If the tip sputters long enough, the electric field strength will drop below the work potential and emission will cease. This mechanism effectively reduces the life span of the device.

To counter these limitations, several FE cathodes were designed that replace the metal cones with another material, one resistant to sputtering and corrosion. Carbon became the material of choice, with some cold cathodes constructed with diamond, or a diamond-like carbon substance. Carbon based cathodes also benefited from a lower work function compared to metal cones. In 1991 micron-long carbon nanotubes were discovered and quickly adapted for use in FE cathodes.<sup>6</sup> These nanotubes offer several advantages in that they were thermally stable, chemically inert, with an electrical conductivity similar to copper yet as strong as steel. One of the biggest advantages of CNTs over metal cones is that as a CNT sputters, it reduces in height while still retaining a sharp tip.

A recent FE cathode that makes use of these advantages consists of a mat of CNTs adhered to the base with a grid above as a gate. Figure 2 shows a scanning electron micrograph of the CNTs in such a mat. Tests of this cathode design show an emission current density of 1 mA/cm<sup>2</sup> at about 2.5 mA/W.<sup>7</sup> An oxidative treatment increases the current emission up to 100 mA/cm<sup>2</sup>. However, this configuration does not constrain each nanotube to the same height, which then causes the emission current density to lack uniformity. In addition, as individual nanotubes sputter and burn up other nanotubes will become active; this in turn causes the emission current density to be unsteady and a function of space and time.

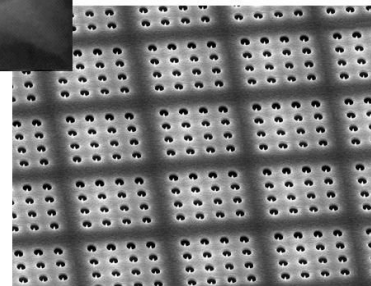
To correct this unsteady emission, yet still retain all the benefits of CNT cathodes, it is proposed to fabricate a carbon nanotube cold cathode (CNCC) such that it

a. Close-up of single tip



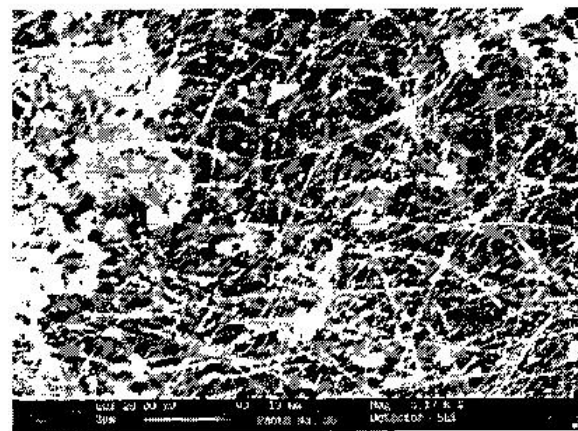
└ 1 μm ┘

b. Portion of 10,000-tip array



└ 10 μm ┘

**Figure 1. Electron micrographs of a Spindt cathode.** View of a) a close up of a single tip and b) the cathode array.<sup>5</sup>



**Figure 2. Electron micrograph of a CNT mat used in FE cathodes.**<sup>7</sup>

consists of many vertically aligned nanotube arrays. A cathode built of these arrays, each with the same height, produces an evenly distributed, steady emission of electrons with a long life-span while remaining chemically inert, and is both thermally and mechanically robust. This design of CNCC is fabricated by Georgia Tech and this paper characterizes the average current density emitted by three CNCCs and further explores the current emission when the gate-cathode voltage is cycled, as well as over time in a 50-hour life test.

## II. Experimental Design

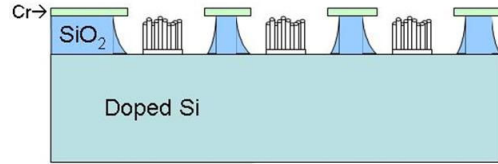
A CNCC in this case should operate in conjunction with a low-power EP device. To this end, the cathode should operate at pressures less than  $10^{-4}$  Torr, supply a current between 0.1 to 1.0 mA, and have a life-span of 1500 hours (the expected life-span of a 200-W HET). The overall design of the CNT cathode follows the pattern developed by Spindt with a few changes to the fabrication process to grow the nanotubes.

In order to meet the set requirements, the cathode is designed with certain materials for each electrical component. The CNT arrays are the cathode with a layer of chromium (Cr) as the gate and a layer of silica ( $\text{SiO}_2$ ) isolating the two. To begin cathode construction, the base of the cathode is made from an n-type doped silicon wafer, as n-type doping provides a greater number of electron donors for emission. A layer of photoresist is spun on the backside of the wafer to prevent oxide growth in order to maintain an electrical connection to the cathode. A thin film of  $\text{SiO}_2$  is thermally grown on the top side of the wafer, followed by a thin film of Cr, deposited by electron beam evaporation. The various cathode arrays are patterned via standard photolithography. After the pattern is developed, the Cr gate is etched away in the shape of the cathode arrays via a standard chromium etch process. After etching the Cr gate, the insulating  $\text{SiO}_2$  is etched via reactive ion etching. Since etching the two materials are both isotropic processes and the insulator is much thicker than the gate, the gate and insulator materials are selected such that the insulator etch would not completely remove the gate material. Furthermore, neither material inhibits CNT nucleation or growth.<sup>8</sup> The etched Cr and  $\text{SiO}_2$  allow a line-of-sight deposition path for the deposition of the iron catalyst layer directly on the n-type Si, insuring an ohmic connection between the cathode and the base. The photo-resist and excess Fe is removed via a stand liftoff process via sonication in acetone. The sample is heated in a quartz furnace with methane, acetylene, and hydrogen to synthesize the nanotube arrays. Figure 3 shows a side view of the original design of the CNT cathode.

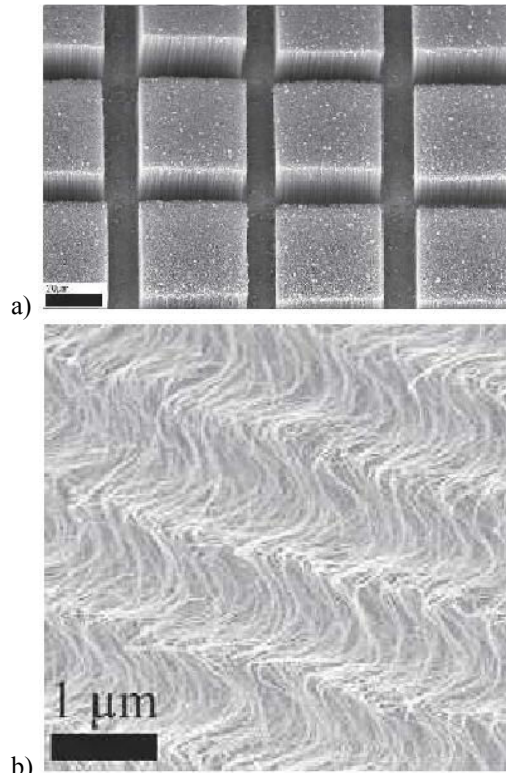
Figure 4 shows micrographs obtained via scanning electron microscopy (SEM) of both the square CNT arrays and a closer view of the morphology. The arrays themselves are aligned normal to the base, with individual height of the nanotubes within 2% of the others within the same array, and each array has only a 5% variation in height in comparison to other arrays. The arrays are arranged into a certain pattern based on the shaping of the chrome slide. These patterns consist of a repeated 2-D shape with a given pitch, or separation distance between the centers of each shape. The patterns of the three samples tested are outlined in Table 1.

**Table 1. Cathode CNT array patterns.**

Sample	Pattern Shape	Pattern Size ( $\mu\text{m}$ )	Pitch ( $\mu\text{m}$ )
408	Ring	8	64
412	Circle	8	32
417	Ring	2	16



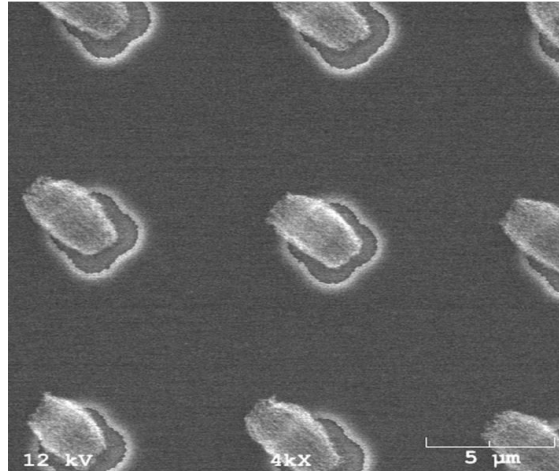
**Figure 3. Diagram of a CNT FE cathode.**



**Figure 4. Micrographs of CNTs. a) Square arrays of carbon nanotubes grown on iron at 700° C, b) CNT morphology.<sup>9</sup>**

Individual nanotubes consist of multi-walled graphite tubes that have an outer diameter between 10-19 nm. Despite the overall structure in the CNT arrays, the individual nanotubes display a kinked vine-like morphology and have a degree of anisotropy.<sup>9</sup> However, this anisotropy is still less than that seen in previous incarnations of CNT configurations.

The design illustrated in Fig. 3 is an example of an intrinsic cathode, one where nanotubes are below the chromium gate. The current process for growing the CNTs creates nanotubes that are too long and thus extend beyond the gate. At the present state this requires the addition of an external gate 1.3 mm from the surface of the original gate, termed an extrinsic triode configuration when paired with an anode. Figure 5 shows an example of such overgrowth in one sample of CNTs in a plus-shaped array. Future work will include a revised fabrication process to reduce CNT length and implement a thicker SiO<sub>2</sub> layer in order to return the setup to an intrinsic configuration.



**Figure 5. Micrograph of overgrown CNTs.**

Once the fabrication process is completed, the CNT samples are subjected to visual inspection using optical microscopy. The presence of CNT growth is conveyed by the appearance of black on the silicon wafer. If the black coloration is present, the sample is further inspected under a SEM to determine the actual CNT growth and shape. Once abundant, well-aligned CNT growth is confirmed the sample is mounted to a stainless steel body and paired with an external molybdenum gate.

In order to characterize an emission current density, the current is normalized by either the average area of the nanotubes, or by the area of the emitter as a whole. For commercially available CNT cathodes these two areas are the same as the nanotubes have no particular arrangement; however, in the GT cathodes, designated Samples 408, 412, and 417, the nanotube arrays only occupy 4% of the emitter surface. The former is a more accurate means to evaluate the effective utilization of the nanotubes, whereas the latter is of more importance in cathode selection in system design. Since the objective is to determine the utilization of the CNTs, current density is calculated in terms of CNT area, which is 0.022 cm<sup>2</sup> for all three samples.

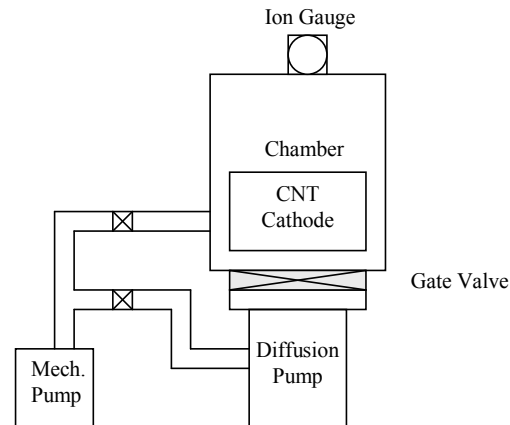
### III. Experimental Apparatus

#### A. Vacuum Facilities

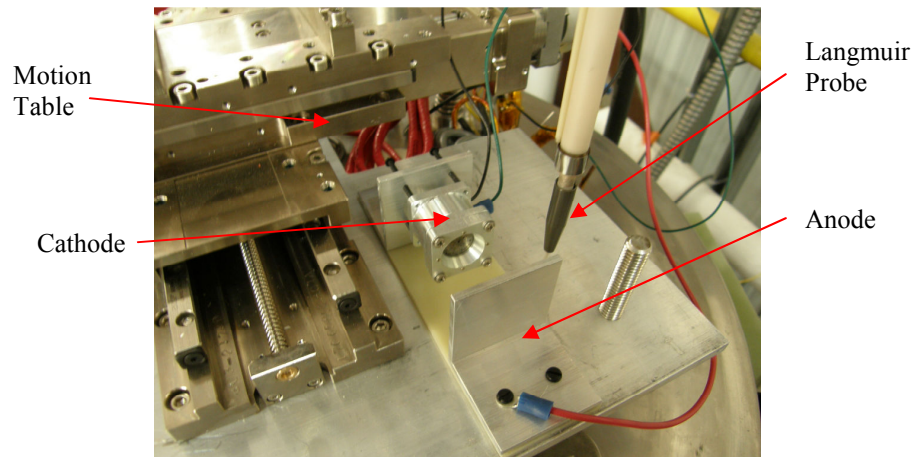
All experiments are conducted at the Georgia Tech High-Power Electric Propulsion Laboratory in a 0.5 m diameter by 0.7 m tall stainless-steel bell jar vacuum system. The chamber is evacuated with a Varian VHS-6 diffusion pump which is mechanically backed by an Alcatel 2033 SD rotary vane pump. An uncooled optical baffle prevents oil from back-streaming into the chamber. The diffusion and backing pumps have pumping speeds of 2400 l/s (for air) and 8.33 l/s, respectively. Pressure in the chamber is measured by a Bayard Alpert 571 ion gauge in connection with a SenTorr ion gauge controller with an overall error of 20%.<sup>10</sup> Figure 6 shows a diagram of the bell jar vacuum system.

#### B. Diagnostics

The CNT base is fitted into a stainless steel mount which is fitted on an aluminum bracket with four 2-56 size nylon machine screws to maintain electrical isolation. Another aluminum bracket is placed 2.5 cm opposite from the face of the cathode body as an anode. While not used in this experiment, Langmuir or Faraday probes are mounted on a 6" 3-axis Orton MX80S motion table system to allow for spatial sweeps of the cathode face. Figure 7 shows a picture of the setup.



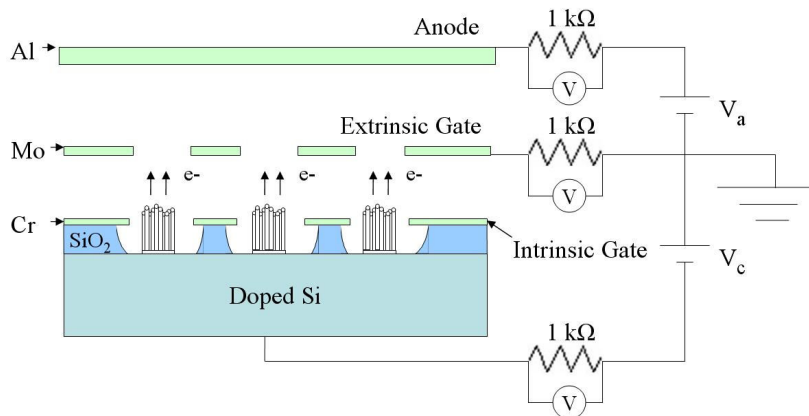
**Figure 6. Diagram of the bell jar vacuum system.**



**Figure 7. Experimental Setup with CNT cathode mounted.**

### C. Experimental Apparatus

The CNT cathode is paired opposite to a stainless steel anode 2.5 cm downstream of the cathode. The cathode, gate, and anode are connected in a triode configuration, as shown in Fig. 8. The anode bias voltage,  $V_a$ , is supplied by a Sorensen DCS60-20E power supply. The cathode bias voltage,  $V_c$ , is supplied by two Sorensen DCS600-1.7E power supplies in series. Both the anode and cathode power supplies share the same ground. The positive of the cathode supply is connected to the ground so that the cathode voltage is biased negative relative to ground. The anode is biased positive relative to ground. The currents to the gate, anode, and cathode are measured across three 1-k $\Omega$  resistor shunts with a Fluke multi-meter. The resistance value has a tolerance of  $\pm 5\%$ <sup>11</sup>, and the multi-meter has an uncertainty of 0.1%.<sup>12</sup> For the life test, the gate resistor shunt is replaced with a 1-M $\Omega$  resistor and the voltage across the shunt is measured with a 1000:1 voltage transformer connected to an Agilent 34970A data acquisition unit with an uncertainty in the voltage of 0.004%.<sup>13</sup>



**Figure 8. Cathode electrical schematic.**

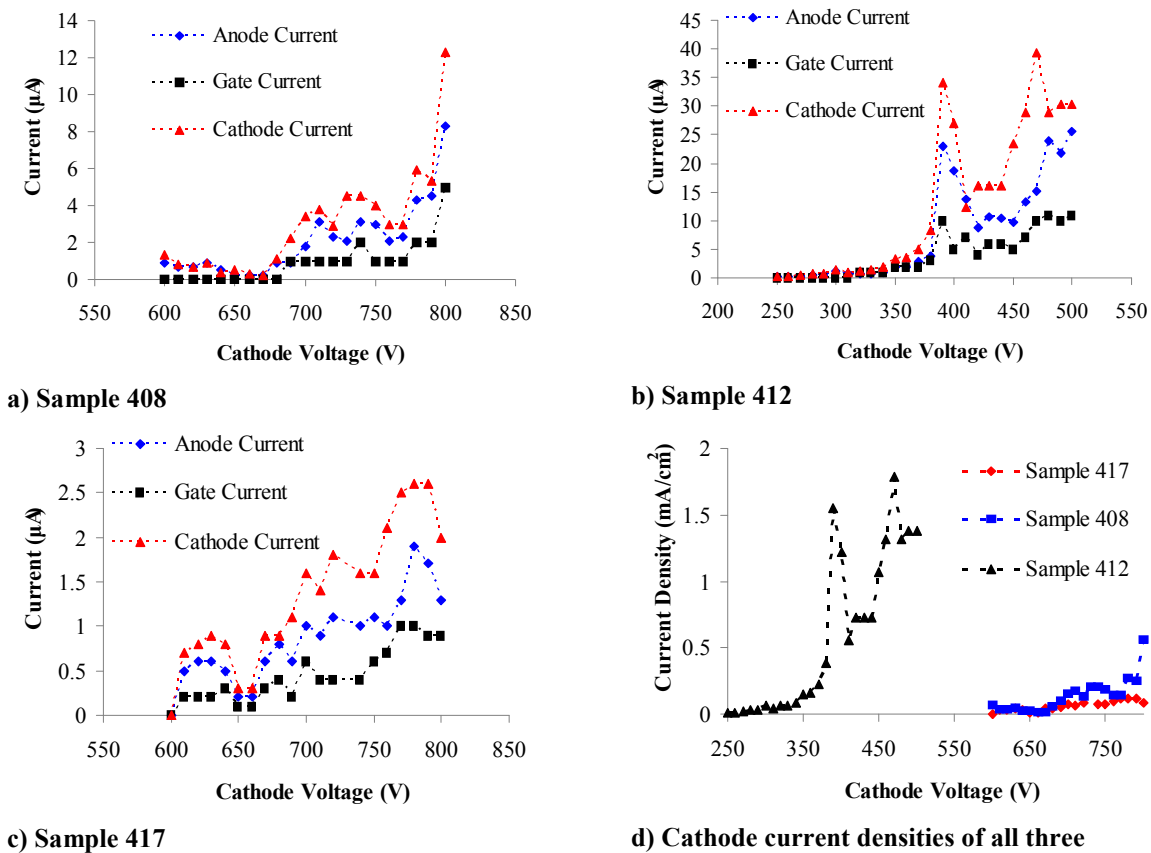
### D. Procedure

Characterization of the cathodes is done in the same manner seen in the literature. The chamber is evacuated to a pressure below  $3 \times 10^{-5}$  Torr. The anode is biased 50 V, and the cathode is biased, with the magnitude of the bias increased until a cathode emission current is measured across the cathode shunt resistor. The cathode voltage is increased in 10 V steps up to 200 V beyond the turn-on voltage, generating an I-V trace of the overall cathode emission. The average current density is calculated as the average current output divided by the area of the emitter occupied by the CNT arrays. The cathode with the highest emission current density is cycled through its operating voltage four times in 10 V steps, with a five minute delay between each point to allow for the nanotubes to stabilize.

The cathode is set to 550 V relative to the gate and the current is measured every 30 seconds over a period of 50 hours.

#### IV. Experimental Results

The transient emissions of the cathodes during start-up are measured as a means to perform an initial comparison. Figure 9 shows the emission current for each cathode in terms the current across each component shunt normalized by the emitter area, with an uncertainty of 5%. It is required that anode current and the gate current together equal the cathode current, as the current collected by the anode and gate should not exceed the current emitted from the cathode. While this requirement exists, there are a few exceptions where the currents change too rapidly to allow for simultaneous measurement of all three shunts. These rapid changes in current emission are caused by a combination of start-up effects, primarily the out-gassing of residual water vapor in the cathode and its subsequent ionization, and the shedding of the outer nanotube walls. These transient events result in a rapid increase in current emission with a short duration time.



**Figure 9.** Current emission for samples a) 408, b) 412, and c) 417. d) Cathode current densities for samples 408, 412, and 417. 50 V anode voltage,  $2.2 \times 10^{-5}$  Torr pressure.

Sample 412 has the highest output current density it is cycled through the cathode voltage range of 350 to 600 V four times in steps of 10 V with a five minute interval between each step. Figure 10 shows, with the exception of the first sweep, that there is a general agreement between each cycle. There is also an increase in current emission between the first sweep and later sweeps as the cathode burns off impurities that are deposited when the cathode is in contact with air. It is seen that there is a decrease in peak current density between Fig. 9 and Fig. 10. When a given voltage applied to the cathode there is an initially unsteady emission, most likely due to nanotubes interacting in the increased electric field. This unsteady emission generally lasts for only a few minutes and results in an

increased current than what is emitted under steady conditions. Thus the emission of sample 412 under cycling with five minute intervals shows the steady emission current density, rather than the initial unsteady behavior.

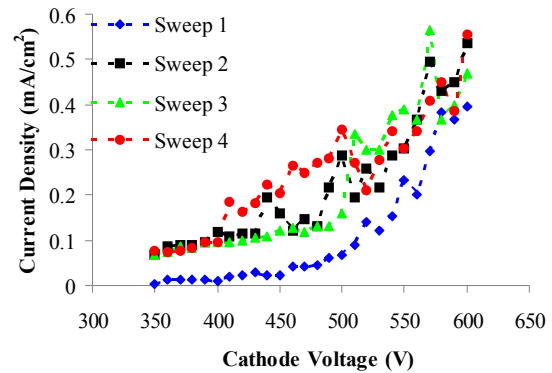
The results of the subsequent 50-hour life test demonstrate the behavior of the CNT cathode over time. Figure 11 shows that while the output current density overall keeps to an average value of  $0.51 \text{ mA/cm}^2$  ( $\pm 5\%$ ), there is a rather large spike in emission current for several hours near the 10<sup>th</sup> hour of the test. There are a few points that are several times larger than the average. Furthermore, only a small percentage of the emission current went into the gate and that a large majority of the current is collected by the anode. This demonstrates a measure of efficiency with little current recycled between the gate and the cathode.

## V. Discussion

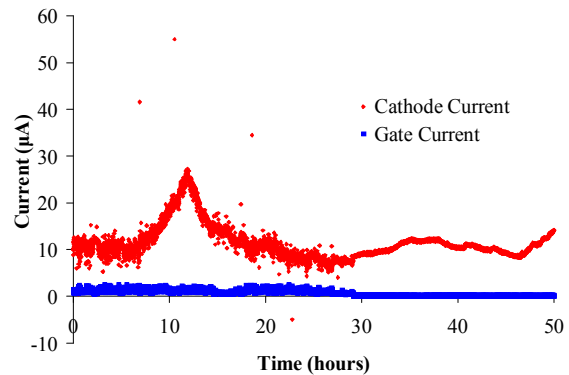
There are several potential causes for the difference in emission currents between the three samples. The primary point of interest is the geometry of the nanotube arrays. In each sample a different pattern is used to arrange the CNT arrays. One possible explanation for sample 412 having a larger emission current density is due to it having a larger pattern size and smaller pitch. However, at the same time if that is the case, sample 412 would also have overall fewer electric field lines converging on any single nanotube, as the electric field is spread over a larger number of CNTs. Another possibility is that the height of the nanotubes in sample 412 is larger than that in samples 408 or 417. Yet, since all three samples are produced in the same process at the same time, the only height difference present is the 5% height variation inherent in the fabrication process. The separation distance between the CNTs and the extrinsic gate screen is over an order of magnitude larger than the height of the CNTs, thus making any increase in local electric field strength from the height variation negligible.

Another point of interest is the difference in turn-on voltage. Both samples 408 and 417 have similar turn-on voltages, whereas 412 began emission at a much lower cathode voltage. A comparison of samples 408 and 417 against sample 412 shows that they have similar current emission. It is possible that both similarities in behavior are due to similarity of CNT pattern shape, as both 408 and 417 are configured in ring patterns. With only a small sampling so far however, it is not certain if this is the case. One means to quantify this idea is to model the electric field behavior for each geometric configuration. Field emission theory states that the emission current density is a function of the electric field strength and the emission material work function. If changing the shape of the emitter pattern from rings to circles increases the local electric field strength, then it stands to reason that is the cause for the change in behavior.

The cycles of sample 412 demonstrate that after the first sweep the cathode performance roughly stabilizes to a repeatable pattern. The initial shift from the first sweep to the second is most likely due to burning off of oxide deposits. These deposits formed during the week prior to the test when the cathode was exposed to air. The minor variations between sweeps 2 through 4, as well as the occasional shifts during a sweep are most likely due to degradation of CNT sections and minor out-gassing of residual water vapor. While the CNTs are arranged in a repeated pattern, the individual nanotubes themselves occasionally shed their outer walls. This process might disrupt the local electric field and thus the individual contribution to emission current.



**Figure 10. Output current density for sample 412.** 10 V steps, 5 minute increments, 50 V anode voltage, pressure  $2.1 \times 10^{-5}$  Torr.



**Figure 11. Life test of sample 412 for 50 hours.** Anode voltage 50 V, cathode voltage 550 V, average pressure  $9.1 \times 10^{-6}$  Torr.

The above mechanism likely occurs over time during the life test, which explains the small peaks and troughs in the shape of the cathode current in Fig. 11. In the 30<sup>th</sup> hour of the test the data becomes noticeably smoother. It is possible that after nearly 30 hours most of the nanotubes have shed their outer walls and are reduced to single-walled nanotubes. Once the CNTs are reduced to a single wall they degrade with a decrease in height, which is expected to have less of an effect on the local electric field than shedding a nanotube wall. The individual outlying points prior to hour 30, with the exception of the point with a negative current, are possibly due to a combination of out-gassing of water vapor and wall shedding. The single point of negative current is most likely due to a diagnostic error.

The cause for the large spike around the 10th hour is unknown. The magnitude of both the duration and increase in current emission is too large to be from single nanotube events such as individual CNT degradation. Furthermore, the emission current density both prior to and after the spike are very similar in both mean and standard deviation. If the spike is caused by some change in nanotube structure, which is inherently irreversible, the current emission should not revert to its previous behavior. Examination of this phenomenon will be continued as more and longer life tests take place. With the exception of the approximately five hour long spike in current, the emission current maintained an approximate average value of 0.51 mA/cm<sup>2</sup> with 92% of the emission current reaching the anode. The emission current to input power performance of the device is 1.7 mA/W. This is to be followed up by a longer duration life test to examine how the cathode performs as it approaches the end of its usable life. There also remains to be seen how to design the cathode to withstand stress due to vibration, which is of great importance if the design is to survive a launch into orbit for deployment.

## VI. Conclusion

In conclusion, three CNT cathodes fabricated from vertically aligned arrays have operated to provide current densities up to 0.6 mA/cm<sup>2</sup> after the initial start-up. One of the cathodes has operated for 50 hours and demonstrated an average emission current density of 0.51 mA/cm<sup>2</sup>. In order to expand on this work, more CNT cathodes of different CNT pattern shape, size, pitch, and appropriate length will be tested to determine the mechanism behind activation voltage and output current. These tests will be compared to computer simulations of the electric field around for that given CNT pattern to develop a correlation between simulated electric field emission and measured current emission. Longer duration life tests are also needed to examine the behavior of CNT cathodes over time frames on the order of the life span of a HET, as well as to determine how the cathodes behave when they are run long enough to fail.

## Acknowledgments

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