

Re-generable Field Emission Cathodes Part II: Quenched Emitter Performance Analyses

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Field emission cathodes have been considered for use as neutralizers on electric propulsion systems. In order to be compatible with EP, the field emission electron sources must be capable of long-life operation and must be robust enough to survive in a wide range of vacuum environments. This paper reports on field-emitters that can achieve long life due to their ability to be re-generated – repairing wear-induced damage. The re-generable emitters were tested against tungsten emitters to compare the re-generable technology with historically proven field emitters. The first type of tungsten emitter used was a DC electrochemically etched tungsten wire with a relatively smooth surface and a single sharp endpoint. The second was a DC and AC electrochemically etched tungsten wire with a roughened surface and a sharp endpoint. The electrochemically etched tungsten field emitters were used to establish electron field emission at pressures of $<10^{-8}$ Torr. They were then exposed to elevated pressure while observing emission. The smooth pure tungsten emitters survived 1 - 2 exposures to increased pressure but required increased extraction voltage to obtain emission with each subsequent test and the roughened pure tungsten emitters lasted 4 - 7 exposures to increased pressure before being permanently damaged. The re-generable emitters were also used to obtain electron emission at a vacuum pressure of $<10^{-8}$ Torr and then the emitters were exposed to elevated vacuum pressure, up to 10^{-5} Torr. In all cases emission from the re-generable emitters lasted 10's of hours longer at increased pressure even before they needed to be re-generated. After re-generation the tips demonstrated the same performance as when new.

I. Introduction

FIELD emission electron sources take advantage of locally enhanced electric fields from nano-scale sharpened electrodes to force electrons to emit from the surface of the electrode into vacuum via a quantum tunneling effect known as Fowler-Nordheim emission. The local electric field enhancement from the emission electrode is inversely proportional to the electrode tip radius so the sharper the emitter tip the lower the electric potential needed to obtain electron field emission. Field emitter tip radii on the order of 1's to 100's of nm are typically employed and are constructed using micro-fabrication techniques¹ from a variety of materials, including tungsten, molybdenum, and carbon nanotubes.²

For years field emission electron sources have been used in flat panel displays, focused electron beams for electron microscopy, and neutralization for spacecraft mass spectrometry.³⁻⁶ For each of these applications, field emission cathodes have demonstrated reliable operation and long lifetimes, mainly due to being operated in pristine ultra high vacuum (UHV) environments. For space applications, the field emitters must be able to survive in weakly ionized plasma environments with pressure that can reach milli-Torr levels. A drawback to the small emitter tip radii that are required for field emitters is that they are fragile and delicate.^{7, 8} When employed in electric propulsion environments the field emitters can and will degrade due to the local plasma environment. Therefore, to implement field emitters with electric propulsion systems it is important to assess their lifetime in elevated pressure environments.

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This paper is the second part of a two-part paper⁹ and is focused on comparing re-generable field emitters with pure tungsten emitters. Tungsten field emitters were chosen as a benchmark due to the extensive characterization of single-needle tungsten emitters historically, and since Spindt-type arrays have been made from refractory metals like molybdenum and tungsten.^{1, 10} Since Spindt-type emitters are made up of thin films of refractory metals, the solid pure tungsten emitters should have had similar (or possibly even better) performance. Investigating the emission characteristics of tungsten emitters for long durations of time and at elevated pressure was done to establish a baseline for the newer re-generable technology. Re-generable field emitters for electric propulsion systems were first demonstrated by Makela and King in 2007.¹¹ The re-generable field emitters were created by operating an indium liquid metal ion source (LMIS) and freezing the sharp ion emitting Taylor cone. The sharp cone that was solidified, or quenched, at the apex of the LMIS was on the order of 1's to 10's of nm and could be used for efficient electron field emission.

II. Goal of Study

The primary goal of this study was to compare the performance of re-generable field emitters to that of solid tungsten emitters – chosen as a benchmark – under conditions comparable to those in the vicinity of an electric propulsion system operating on a spacecraft. For this study, a series of experiments were performed to observe the emission characteristics of each emitter for long durations of time in an ultra high vacuum (UHV) environment. Furthermore, each type of tungsten emitter and the re-generable emitters were tested while increasing the vacuum pressure from a UHV environment to $> 10^{-5}$ Torr to determine how long and to what pressure emission could sustain. Each field emitter was exposed to elevated pressure conditions multiple times to determine their ability to tolerate cyclic damage.

III. Experimental Apparatus

A schematic of the apparatus is shown in Figure 1. As shown, a loop of tungsten wire was fixed to a Teflon block and the emitter tip was placed < 0.5 mm away from the extraction electrode. A micro-ammeter was placed in-line with the high voltage extraction electrode to measure the emission current. All of the components were constructed from Teflon, stainless steel, and tungsten.

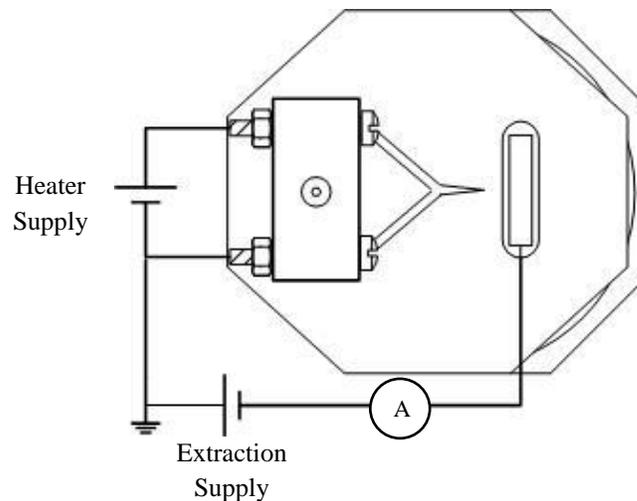


Figure 1. Electrical schematic of the FE-SEM specimen fixture.

All field emitters used in this study were formed by electrochemically etching tungsten wires to form a sharp tip. Two emitters, referred to as 'bare' tungsten emitters, were uncoated after etching and thus the current was emitted from the tungsten tip. The 're-generable' emitters were created by first electrochemically etching tungsten needles in an identical process, however the etched needles were then coated in indium. Indium nanotips were formed at the emitter apex using a process described by Makela.¹²

Two bare tungsten emitters were tested. The first bare emitter tested was DC etched in a 2M NaOH solution to create a sharp and smooth emitter tip, as shown in Figure 2.¹³ The tungsten emitter was etched to have similar geometry to state-of-the-art Spindt-type field emitters. The second type of bare emitter that was tested was DC and then AC electrochemically etched in a 2M NaOH solution to create a roughened surface structure as shown

in Figure 3.¹⁴ The roughened structure was intended to create an emitter with surface grooves, exactly the same as the field emitter that was coated with indium for the re-generable experiments. A possible indirect benefit of adding surface grooves was that the number of field-emitting nano-structures on the surface may have also increased.

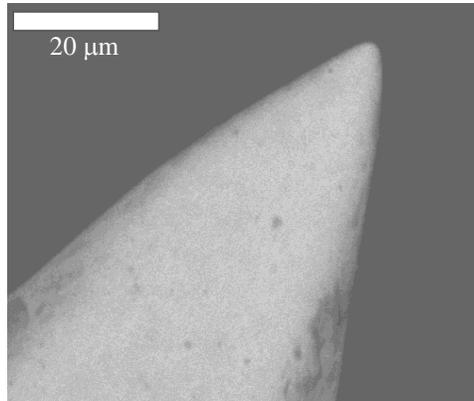


Figure 2. DC electrochemically etched bare tungsten emitter.

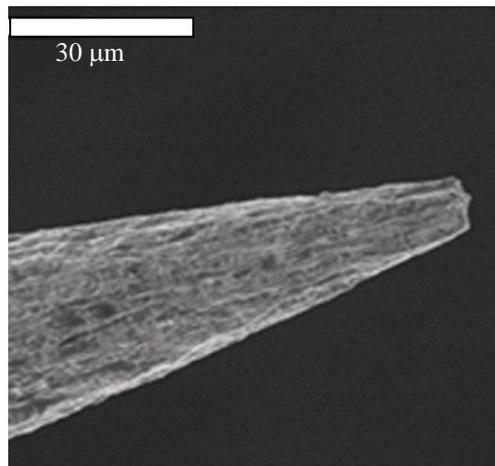


Figure 3. DC and AC electrochemically etched bare tungsten emitter.

The Ultra High Vacuum chamber is approximately 0.5 meters in diameter by 0.5 meters long. The chamber has a base pressure of 10^{-9} Torr, which is achieved by pumping with a single 280-L/s turbo-molecular pump that is backed by a 110-l/min dry scroll pump. The chamber is also equipped with a 300-L/s combination ion/sublimation pump that can maintain pressure of 10^{-9} Torr. An operating pressure of $< 10^{-8}$ Torr was maintained throughout lifetime experiments.

IV. Experimental Procedure

To establish electron emission from the bare tungsten emitters, the extraction electrode was biased positively with respect to the emitter (which was at ground potential) and the extraction voltage was increased until emission was acquired. The emission current was recorded using a micro-ammeter that was placed in-line with the extraction supply. For lifetime studies the extraction voltage was held constant and emission current was observed while at vacuum pressure $< 10^{-8}$ Torr. For the elevated pressure experiments emission was first established at vacuum pressure $< 10^{-8}$ Torr for 2 to 24 hours and then the ion pump was turned off to let the vacuum pressure slowly increase while observing the electron emission current. Once emission ceased the extraction voltage was decreased and the ion pump was turned back on to reduce the vacuum chamber pressure. When the vacuum pressure returned to $< 10^{-8}$ Torr the same procedure was used to establish emission and expose the operating electron source to elevated vacuum pressure. The process was repeated until emission could no longer be achieved at reasonable

extraction voltages. Ultimately, exposing the emitter tips to increased pressure multiple times would render the tip useless as an electron emitter.

For the re-generable emitter experiments, the same method discussed in Part I⁹ of this series of papers was used to re-generate the emitters. The procedure began with establishing ion emission by first heating the emitter, shown previously in Figure 1, to 2.75 A, 1.3 V with the purpose of maintaining the indium-coated electrode above the melting temperature of indium, which is 156.6°C. The high-voltage extraction supply was then used to increase the potential between the emitter and the extraction electrode to obtain ion emission. For each set of experiments the extraction voltage was increased until the desired ion emission current was reached and then the extraction electrode voltage was held constant for one minute. Leaving the extraction electrode constant, the heater power was turned off to quench the operating LMIS. After the LMIS was allowed to cool for one minute, the extraction supply was also turned off – leaving sharpened nano-structures to use for electron emission. The re-generable emitters were then tested using the same procedure as the bare tungsten emitters.

V. Experimental Results and Discussion

A. Smooth Bare Tungsten Emitters

The first experiment was performed with a smooth bare tungsten field emitter, as shown previously in Figure 2. The emitter was electrochemically DC etched using the procedure described by Makela et al.¹⁵ The smooth bare tungsten emitter was tested in a UHV chamber at a vacuum pressure of 10^{-9} Torr. Prior to lifetime testing, an electron I-V curve was acquired by sweeping the extraction voltage while recording the emission current so that the data could be used for Fowler-Nordheim analysis. The Fowler-Nordheim model was then used to estimate the emitter tip radius. For the smooth bare tungsten emitter, the model yielded a tip radius estimate of 13.9 ± 1.2 nm. Electron emission was then achieved by increasing the extraction electrode to 1.4 kV to obtain an emission current of 3 μ A. The extraction voltage was then held constant and emission current was observed for 625 hours. To conclude the experiment, at $t_e = 625$ hours (chosen somewhat arbitrarily based on laboratory schedule constraints) the ion pump on the UHV chamber was turned off to increase the chamber pressure. At vacuum pressure of 10^{-7} Torr, and after less than two minutes, the emission ceased, as shown in Figure 4. A Fowler-Nordheim analysis was not performed after the 625 hour test because electron emission could not be acquired despite increasing the extraction voltage to 4 kV at UHV conditions.

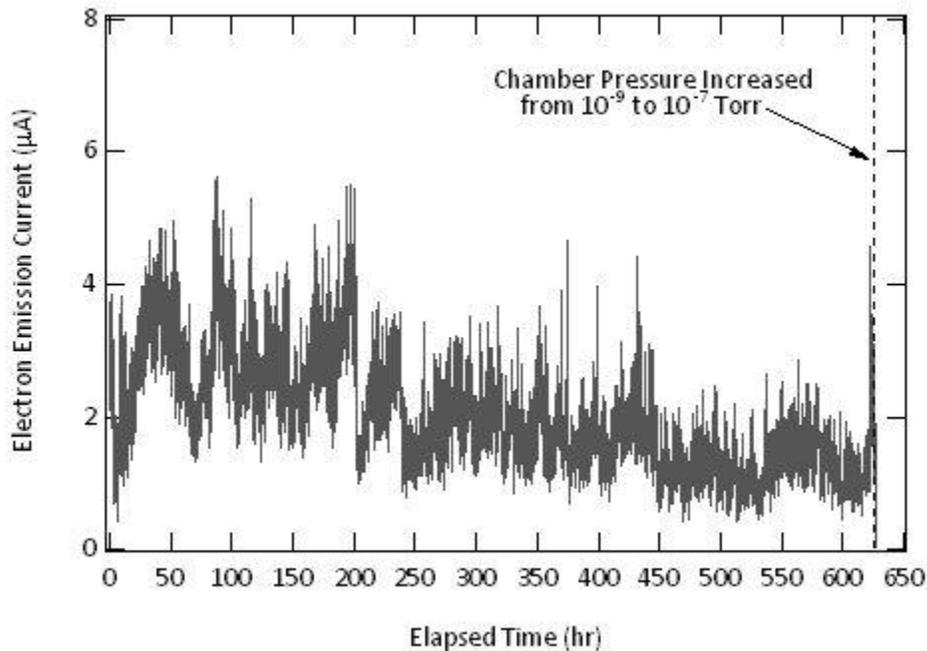


Figure 4. Electron emission current from an electrochemically etched bare tungsten field emitter, showing a sudden decrease in emission current when background pressure was increased from 10^{-9} to 10^{-7} Torr. Emission current could not be established in subsequent testing (at an extraction voltage of 4 kV) indicating that the smooth bare tungsten emitter tip had been permanently damaged when exposed to an increased vacuum chamber pressure.

After the emission ceased, electron emission could not be re-established at 4 kV. The maximum limit of 4 kV was arbitrarily chosen as an upper bound for the onset voltage for all of the experiments reported. Since emission couldn't be established, the emitter tip must have been catastrophically damaged by the increase in vacuum pressure. Exposure to elevated pressure rendered the bare tungsten emitter useless as a field emission electron source. A second smooth bare tungsten field emitter was tested under similar conditions and was permanently damaged after two exposures to similar vacuum conditions.

B. Roughened Bare Tungsten Emitters

Two roughened bare tungsten emitters were tested for comparison. The roughened emitters were electrochemically DC and AC etched following the procedure described by Makela et al¹⁵ to create surface grooves, as shown previously in Figure 3. Etching the surface grooves on the emitter was intended to make the electrode as close as possible to the indium-coated re-generable electrodes. An electron I-V sweep was acquired on the new roughened bare tungsten emitter at 10^{-8} Torr. Applying the Fowler-Nordheim model to the electron emission I-V sweep yielded a tip radius estimate of 4.6 ± 0.9 nm. After performing an electron I-V sweep, the extraction voltage was adjusted to establish electron emission.

The roughened bare tungsten emitter was then operated for about 20 hours to ensure the emission current was stabilized. Holding the extraction voltage constant, at $t_e = 21.2$ hours the vacuum chamber pressure was increased by turning off the ion pump. As the vacuum pressure increased the electron emission current was observed. Electron emission sustained for about 7 hours at background pressure ranging between 10^{-5} and 10^{-3} Torr, as shown in Figure 5, which was about 7 hours longer than emission sustained using smooth bare tungsten emitters. The background pressure was recorded until the ion gauge turned off due to an overpressure. Unfortunately, the vacuum chamber pressure couldn't be recorded for the duration of some of the tests due to a lack in overlap of measuring range between the ion gauge and thermocouple gauge on the UHV system. Extrapolating the pressure data and analyzing data from other elevated pressure experiments, an approximate vacuum pressure of 5×10^{-5} Torr was estimated when emission ceased.

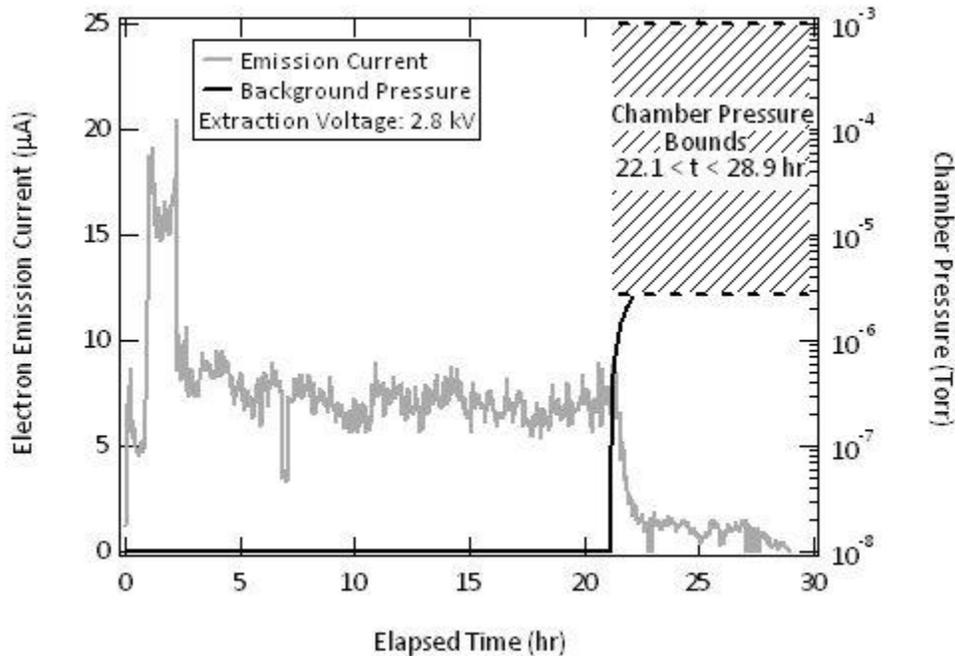


Figure 5. Electron emission current from a roughened bare tungsten emitter held at a constant extraction voltage of 2.8 kV from $t_e = 3.1$ hr to $t_e = 28.9$ hr as background pressure was increased for the first time. The region of unknown pressure was due to a lack of overlap in the cold cathode and thermocouple pressure gauges that were equipped on the vacuum facility.

Once emission ceased, the vacuum pressure was decreased to 10^{-9} Torr and another electron I-V sweep was acquired. The electron I-V sweep taken prior to testing and after the first exposure to elevated pressure are both shown in Figure 6. The Fowler-Nordheim model was applied to the I-V sweep and yielded a tip radius estimate of

9.9 ± 2.0 nm, which was an increase in the emitter tip radius that was observed before exposing the operating field emitter to an increase in vacuum pressure.

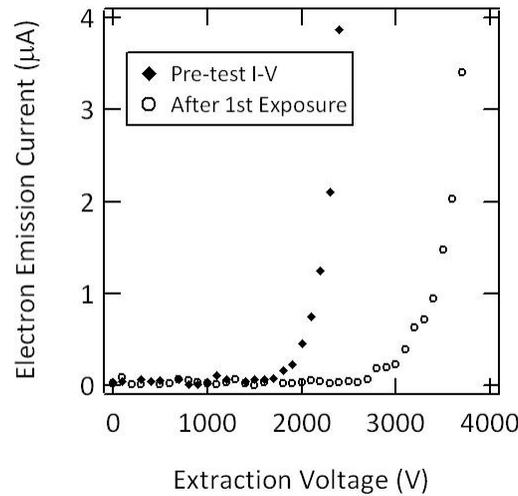


Figure 6. Electron I-V sweep taken from a new roughened bare tungsten field emitter and an electron I-V sweep acquired after the first exposure of the emitter tip to elevated vacuum pressure. The Fowler-Nordheim model was applied to the I-V sweep after the first exposure and the tip radius was estimated at 9.9 ± 2.0 nm.

After the Fowler-Nordheim sweep was taken, electron emission was again established with the roughened bare tungsten emitter at high vacuum. Emission was sustained at constant voltage for about 4 hours at a vacuum pressure of 10^{-9} Torr and then the vacuum pressure was increased while emission was observed. During the first 4.5 hours of the experiment, the emission current gradually decreased when vacuum pressure was constant in the 10^{-9} Torr regime, implying degradation of the tip. After 1.5 hours, emission ceased when vacuum pressure reached 2×10^{-5} Torr. Each exposure will be described in detail in the following paragraphs and the elevated pressure experiments and Fowler-Nordheim analyses will be summarized in Table 1.

During the second exposure to elevated pressure, emission from the roughened bare tungsten emitter lasted longer than electron emission from a smooth bare tungsten emitter. After emission ceased the vacuum pressure was decreased back to 10^{-9} Torr and an electron I-V sweep was performed to apply the Fowler-Nordheim model. The model yielded a tip radius estimate of 6.3 ± 1.3 nm, which was sharper than the estimated tip radius before exposing the emitter to increased pressure. A few possible reasons for a decrease in the tip radius after exposure to elevated pressure are discussed at the end of this section.

The roughened bare tungsten emitter tip was then used to achieve electron emission a third time at high vacuum. Emission was established at an extraction voltage of about 2.1 kV and the electron emission was then left to stabilize for about 4 hours. At $t_e = 3.8$ hours vacuum pressure was increased and the emission current was observed. The emission sustained until $t = 6.5$ hours into the pressure range of 10^{-6} to 10^{-3} Torr. Extrapolating the pressure data, the pressure when emission ceased was approximately 6.3×10^{-6} Torr.

After emission ceased the vacuum chamber pressure was restored to 10^{-9} Torr and an electron I-V sweep was taken and the Fowler-Nordheim model was applied. The F-N model yielded a tip radius estimate of 4.6 ± 1 nm. Since numerous I-V sweeps were acquired and the Fowler-Nordheim model yielded emitter tips that were approximately the same size, it is possible that the roughened bare tungsten emitter may have multiple emission sites with similar nano-structures, as shown in the micrograph previously in Figure 3. Each time the roughened bare tungsten emitter was exposed to increased vacuum pressure, it is possible that a single emission site was damaged but the next sharpest nano-structure could be used for subsequent testing.

For the fourth time, electron emission was established from the roughened bare tungsten emitter tip that had been exposed to increased pressure. Emission current was stable around 4 μ A at constant voltage for about an hour and then the vacuum pressure was increased at $t_e = 1$ hr. Emission current sustained for about 5 hours at increased vacuum pressures ranging from 10^{-5} and 10^{-3} Torr. Extrapolating the pressure data, the vacuum chamber pressure when the emission failed was approximately 2×10^{-5} Torr.

Once emission ceased, an electron I-V sweep was acquired and the Fowler-Nordheim model was applied – yielding an emitter tip radius of about 7.1 ± 1.4 nm. Although the emitter tip radius estimations were within 10 nm of each other, the extraction voltage required to achieve electron emission after successive exposures to increased

vacuum pressure had been increasing and decreasing randomly, indicating that the emitter tip was being damaged after some of the exposures and was getting sharper during other exposures to increased pressure which will be discussed later.

After the Fowler-Nordheim sweep, the extraction voltage was increased to 4 kV to obtain electron emission a fifth time. The initial emission current was 4 μA but quickly increased to about 6 μA . After staying constant at 6 μA for about two hours the vacuum pressure was increased. Emission sustained for about 20 hours at increased pressure. When emission ceased the vacuum pressure was decreased to 10^{-8} Torr and an electron I-V sweep was performed. The Fowler-Nordheim model was applied and resulted in an emitter tip radius of about 10.0 ± 2.0 nm. Thus far the roughened bare tungsten emitter tip was exposed to an increase in vacuum pressure five times and the estimated emitter tip radii have only varied by 5-6 nanometers.

After the tip estimate was calculated the extraction voltage was increased to 3.9 kV at high vacuum and the emission current was observed while exposing the roughened bare tungsten emitter to increased vacuum pressure for the sixth time. Emission lasted for about 4 hours as the pressure was increased. When emission ceased the vacuum pressure was returned to 10^{-8} Torr and another I-V sweep was performed, resulting in a Fowler-Nordheim emitter tip radius estimate of 7.8 ± 1.6 nm. Again, the emitter tip radius estimate was on the same order as the past couple of Fowler-Nordheim analyses.

After the Fowler-Nordheim analysis the same roughened bare tungsten emitter tip was exposed to increased vacuum pressure a seventh time. The emission current sustained for about 5 hours. Emission ceased at about 10^{-5} Torr. After emission ceased, the vacuum chamber pressure was decreased to 10^{-8} Torr and an electron I-V sweep was acquired. A post-test Fowler-Nordheim analysis yielded an emitter tip radius estimate of 14.0 ± 2.8 nm. After seven exposures to elevated vacuum pressure conditions, emission from the roughened bare tungsten emitter tip could not be established at an extraction voltage of 4 kV in high-vacuum conditions.

Table 1. Summary of data from the roughened bare tungsten field emitter exposure to elevated pressure. The field emitter survived 7 exposures to elevated pressure conditions before permanent failure.

Exposure Number	Δt from UHV to Failure hour	Fowler-Nordheim Radius Estimate nm	Approximate Vacuum Pressure at Failure Torr
Pre-test	-	4.6 ± 0.9	-
1st	7	9.9 ± 2.0	5.0×10^{-5}
2nd	1.5	6.3 ± 1.3	2.0×10^{-5}
3rd	3.7	4.6 ± 1.0	6.3×10^{-6}
4th	5	7.1 ± 1.4	2.0×10^{-5}
5th	20	10.0 ± 2.0	-
6th	4	7.8 ± 1.6	8.0×10^{-6}
7th	5	14.0 ± 2.8	10^{-5}

To summarize, the first roughened bare tungsten emitter that was tested survived 7 exposures to elevated vacuum pressure within the test conditions of using a maximum extraction voltage of 4 kV to establish electron emission. Also, the first roughened bare tungsten emitter sustained between 1.5 and 20 hours of electron emission after the increase in pressure began for each experiment. A second roughened tungsten field emitter was fabricated and tested in an identical fashion; this emitter survived 4 exposures to elevated pressure and emission sustained from 1.4 to 20 hours while pressure was increased. The maximum pressure that was measured before emission ceased was 9.9×10^{-5} Torr. Both roughened emitters demonstrated longer lasting emission and survived more exposures to elevated pressure than the smooth bare tungsten emitters, as summarized at the end of Section V.C in Table 2.

C. Re-generable Field Emitters

To investigate the lifetime of re-generable emitters, a dual ion/electron source fabricated according to the technique described in Makela¹⁵ was placed in the UHV chamber and the vacuum chamber pressure was reduced to 10^{-9} Torr. The emitter was then heated and operated at 20 μA of ion emission current for one minute and then

quenched to preserve a solid indium nanotip at the apex. The polarity of the extraction electrode was then reversed to achieve electron emission and the extraction electrode voltage was increased to obtain electron emission at $4\ \mu\text{A}$. The voltage required for $4\ \mu\text{A}$ of electron emission current was $4.1\ \text{kV}$; the extraction electrode was then voltage-limited at $4.1\ \text{kV}$ for the remainder of the experiment. The re-generable field emitter was tested at $4\ \mu\text{A}$ to subject the emitter to the same current as the tungsten field emitters.

Within the first hour of the lifetime test, the emission current increased to approximately $11\ \mu\text{A}$ for a few minutes and then quickly decreased down to about $3\ \mu\text{A}$ as shown in Figure 7. The emission current at constant voltage fluctuated between about 2 and $4\ \mu\text{A}$ after the first hour until about $t_e = 600$ hours. At 600 hours into the experiment the emission quickly increased to about $5\ \mu\text{A}$ and then slowly decreased down to about $3\ \mu\text{A}$ again. At $t_e = 950$ hours of operation the experiment was voluntarily ceased for 5 hours to observe if the emission current would return to the same magnitude when turned back on. The experiment was paused by simply decreasing the extraction voltage from $4.1\ \text{kV}$ to $0\ \text{kV}$.

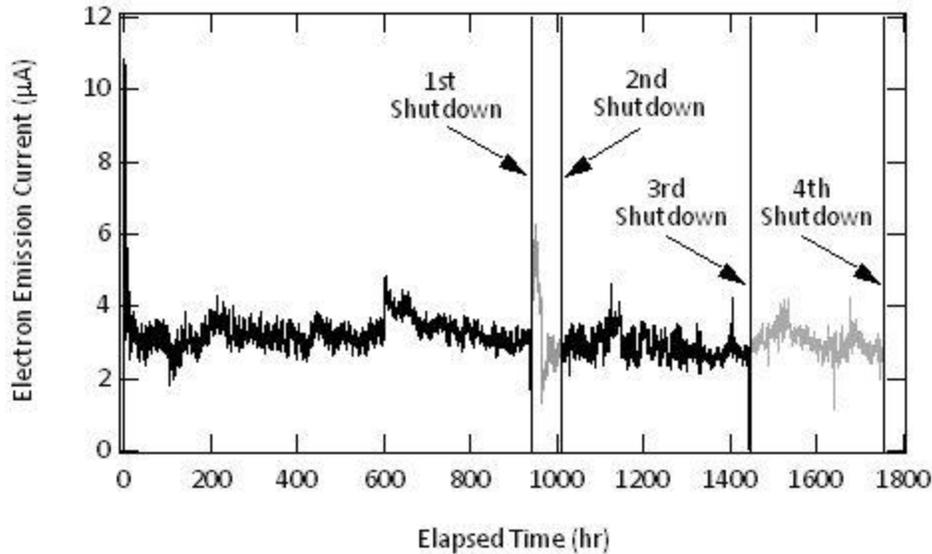


Figure 7. Electron emission lifetime experiment from a single-needle re-generable emitter quenched at ion emission current of $20\ \mu\text{A}$, showing locations of voluntary shutdowns. The extraction voltage was $4.1\ \text{kV}$ and the vacuum pressure was $\sim 10^{-9}$ Torr throughout the experiment.

The experiment was paused for 5 hours and then the extraction electrode was increased back to the original setpoint of $4.1\ \text{kV}$. The emission current increased to almost $10\ \mu\text{A}$ after the onset of emission but quickly decreased to about $5\ \mu\text{A}$ at $t_e = 952$ hours. Then the electron emission current remained at $5\ \mu\text{A}$ until approximately $t = 975$ hours and then rapidly decreased to $2\ \mu\text{A}$. Once reaching $2\ \mu\text{A}$ the emission current slowly increased up near $3\ \mu\text{A}$ until $t_e = 1025$ hours. The experiment was then voluntarily paused again for a span of 4 hours. During the 4 hour period of downtime, an electron I-V sweep was taken to estimate the emitter tip radius. Applying the Fowler-Nordheim model to the I-V data yielded a tip radius estimate of $9.5 \pm 1.9\ \text{nm}$.

After this second shutdown, the extraction voltage was increased back to the setpoint of $4.1\ \text{kV}$. As the extraction voltage was increased the electron emission current increased to $3\ \mu\text{A} \pm \mu\text{A}$ for 435 hours of operation, bringing the total duration of the lifetime test thus far to 1460 hours. At $t_e = 1460$ hours, the experiment was voluntarily interrupted again to obtain another electron emission I-V sweep. The I-V sweep was taken to determine if any change in tip radius was apparent from the first I-V sweep. The Fowler-Nordheim model was applied to the electron I-V data acquired during the third shutdown and yielded a tip radius estimate of $16.4 \pm 3.3\ \text{nm}$.

Following the I-V sweep, the extraction voltage was again increased to $4.1\ \text{kV}$. The emission current gradually increased back to near $3\ \mu\text{A}$ as the extraction voltage was increased. The lifetime experiment was continued for 300 additional hours, bringing the total lifetime test to about $t_e = 1750$ hours. The lifetime experiment was ended after 1750 hours due to the necessity to use the UHV chamber to perform other experiments. To end the lifetime experiment the extraction voltage was turned off and then a final electron emission I-V sweep was taken to estimate the nano-structure tip radius. The Fowler-Nordheim plot was applied to the electron I-V data from the fourth sweep and yielded a tip radius estimate of $16.2 \pm 3.2\ \text{nm}$.

To compare with the single needle bare tungsten field emitters that were exposed to increased vacuum pressure, the same elevated vacuum pressure experiments were performed with the re-generable emitters. The re-generable emitter was first operated as an ion source for 1 min at 20 μA , while heating the emitter with 2.25 A at 0.3 V. After 1 min the heater was turned off and the ion emission was quenched. The extraction voltage was then reversed to obtain electron emission and a Fowler-Nordheim analysis was performed on the ‘New’ emitter at 10^{-8} Torr, as shown in Figure 11. The Fowler-Nordheim model resulted in a tip estimate of about 13.7 ± 2.8 nm.

After the I-V sweep, the extraction voltage was increased to 3.8 kV to obtain electron emission. During the first 20 min of operation at 10^{-8} Torr the emission current had increased to about 25 μA so the extraction voltage was decreased to 2.1 kV in an attempt to keep the emission current around 4 μA . After the emission current remained relatively constant for approximately 1 hour the ion pump on the UHV chamber was turned off to increase the background pressure. The data that were recorded are shown in Figure 8, along with a bound of the possible background pressure. Extrapolating the pressure data, the vacuum chamber pressure was estimated at 8×10^{-5} Torr. As shown, electron emission from the quenched re-generable emitter sustained for over 15 hours.

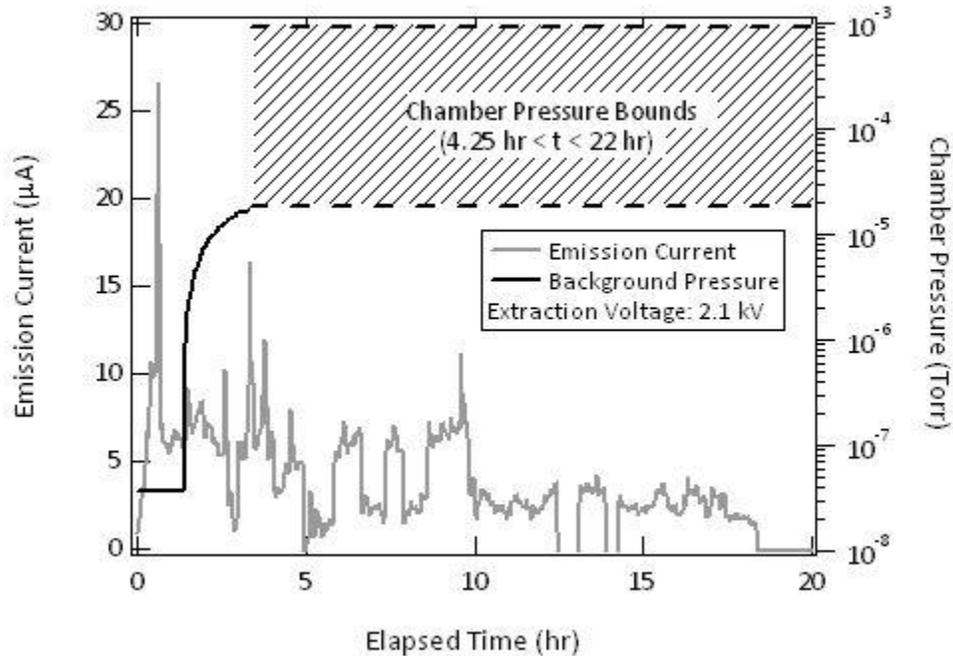


Figure 8. Electron emission current from a quenched re-generable emitter held at a constant extraction voltage of 2.1 kV from $t_e = 0.3$ hr to $t_e = 20$ hr as background pressure was increased.

Once emission ceased, the ion pump on the vacuum facility was turned back on and vacuum pressure was decreased to 10^{-8} Torr. An electron I-V sweep was performed on the emitter to obtain an estimate of the nano-structure radius that was emitting electrons, as shown in the ‘New + 1 Exposure’ data in Figure 11. The Fowler-Nordheim model resulted in a tip radius estimate of 12.8 ± 2.6 nm.

The extraction voltage was then increased to 6.1 kV to obtain an electron emission current of about 4 μA . The extraction voltage was adjusted during the first 30 minutes because the emission current kept increasing. At $t_e = 30$ minutes the extraction voltage was set to 3.4 kV to return the emission current to about ~ 5 μA . The extraction voltage was held at 3.4 kV for the remainder of the experiment, which was an increase of 1.3 kV in the extraction voltage that was necessary to obtain the same magnitude of emission current as before the emission ceased. Once the emission current remained stable for 2 hours, the ion pump on the UHV system was turned off again to increase the background pressure. Electron emission current was maintained for nearly 50 hours after turning off the ion pump and increasing the vacuum pressure from 10^{-8} Torr to between 10^{-5} and 10^{-3} Torr, as shown in Figure 9.

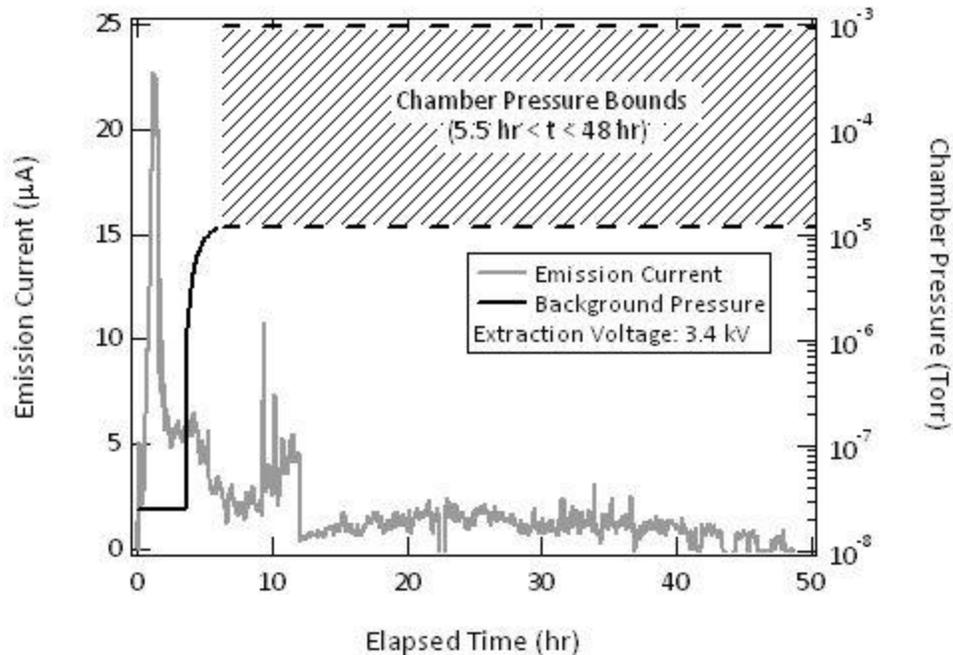


Figure 9. Electron emission current from the same quenched re-generable emitter used in the test of Figure 8. The emitter was held at a constant extraction voltage of 3.4 kV from $t_e = 0.5$ hr to $t_e = 50$ hr as background pressure was increased.

After electron emission ceased, the ion pump on the UHV chamber was turned back on and the chamber pressure was decreased back to 10^{-8} Torr. An electron I-V sweep was acquired, as shown by the 'New + 2 Exposures' data in Figure 11. The Fowler-Nordheim model was applied to the sweep and resulted in a tip radius estimate of about 15.5 ± 3.1 nm. The emitter tip after the first exposure was sharper than the emitter tip after the second exposure, which was expected.

After emission ceased, the vacuum pressure was restored to 10^{-8} Torr and electron emission couldn't be achieved at an extraction voltage of up to 4 kV. The tip must have sustained damage from the two consecutive exposures to elevated background pressure, so the field-emitting nanostructures were re-generated through a sequence of heating, ion-emission, and quenching. The tip was operated at an ion emission current of 20 μ A at 10^{-9} Torr for 1 min while supplying 2.25 A and 0.3 V of heater power. The emitter tip was then quenched and an electron I-V sweep was acquired, as shown by the 'Re-generated' data in Figure 11. The Fowler-Nordheim model was applied to the electron I-V sweep and resulted in a re-generated nano-structure radius of about 14.3 ± 2.9 nm, which was sharper than the emitter tip before re-generating it.

The freshly re-generated emitter tip was then operated as an electron source for over 150 hours at a background pressure of 10^{-9} Torr to demonstrate reliable operation from an emitter tip that had been exposed to increased background pressure multiple times and then had been re-generated, as shown in Figure 10. The extraction voltage was held at 4.1 kV from $t_e = 0$ to $t_e = 1.5$ hr and then the extraction voltage was decreased to 2.5 kV due to a rapid rise in emission current. The extraction voltage was held at 2.5 kV for the remainder of the experiment.

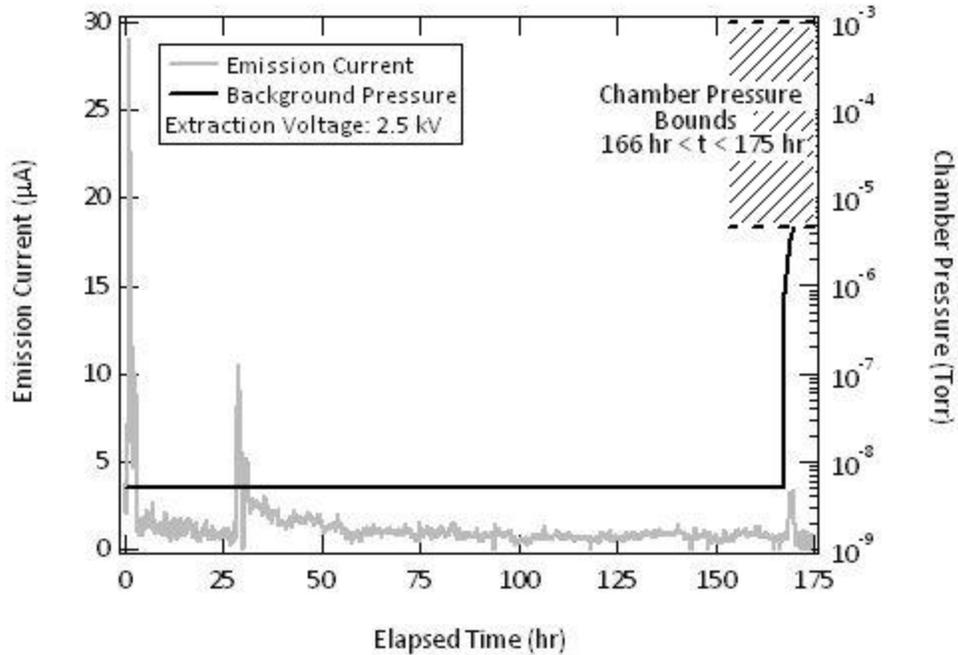


Figure 10. Extended duration experiment for an electron emitter that was re-generated by heating, acquiring ion emission, and then re-quenching after being damaged in the test sequences of Figure 8 to Figure 9. The extraction voltage was held constant at 2.5 kV from $t_e = 1.5$ hr to $t_e = 175$ hr and the test was ended by increasing the vacuum chamber pressure after over 150 hours of operation.

At $t_e = 166$ hr, the ion pump on the UHV chamber was turned off and the emission current was observed as tank pressure increased. At $t_e = 175$ hr, the emission ceased. Once electron emission ceased, the ion pump on the UHV chamber was turned back on and vacuum pressure was decreased to 10^{-9} Torr. A final electron I-V sweep was performed, as shown by the ‘Re-generated + 1 Exposure’ data in Figure 11, resulting in a nano-structure radius estimation of 9.7 ± 1.9 nm after applying the Fowler-Nordheim model. Exposure to elevated pressure should have increased the emitter tip radius, however, emitter tip degradation didn’t occur in this case. Rarely, emitter tip performance increased after exposure to elevated pressure. Possible reasons for enhanced performance after exposure to elevated pressure are discussed at the end of Section V.C.

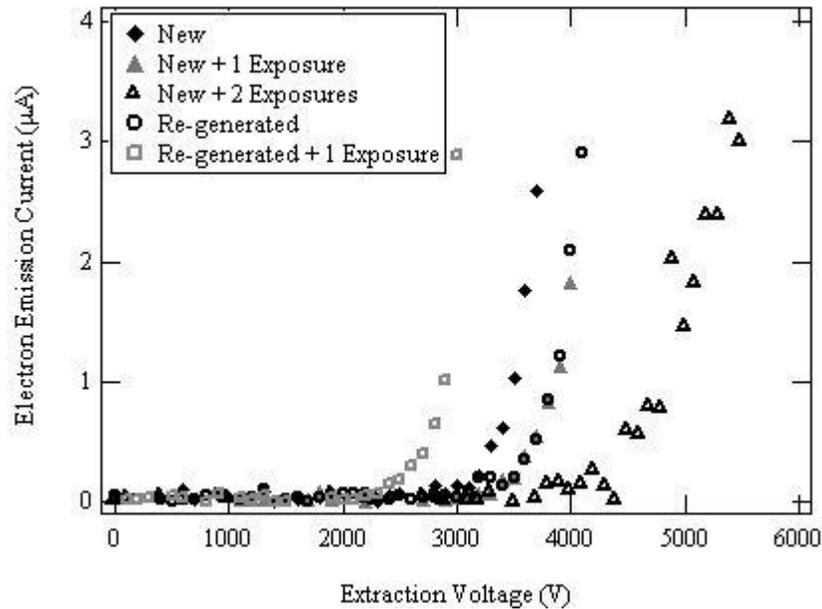


Figure 11. Fowler-Nordheim sweeps taken during each test sequence of operating a re-generable field emitter in UHV conditions and then exposing the emitter to an elevated vacuum pressure environment. After two exposures to elevated pressure conditions the emitter tip was re-generated.

The results from the comparison of the smooth bare tungsten, the roughened bare tungsten, and the re-generable emitter are summarized in Table 2. The table includes the length of time that emission could sustain at elevated pressure, the number of times the emitter was exposed to an increased vacuum pressure before permanent failure was observed, and the magnitude of the maximum vacuum pressure that was recorded while emission sustained.

Table 2. Emitter tip comparison table displaying how long the emitter tips lasted at elevated pressure, how many exposures to increased pressure caused permanent failure, and the maximum vacuum chamber pressure that emission could sustain.

Emitter Type	# of Tested Emitters	Emission Duration at Elevated Pressure	# of Exposures Before Failure	Maximum Tank Pressure Reached
Smooth Bare Tungsten	2	1 - 5 min	1 - 2	10^{-7} Torr
Roughened Bare Tungsten	2	1.4 - 20 hr	4 - 7	10^{-5} Torr
Re-generable Emitter	2	6 - 45 hr	Unlimited [§]	10^{-3} to 10^{-5} Torr

The series of experiments performed with a re-generable emitter demonstrated longer-life operation than single bare tungsten field emitters at increased vacuum pressures. While the quenched re-generable emitter required an increase in extraction electrode voltage after each exposure to increased vacuum pressure, the re-generable emitters survived more harsh conditions than bare tungsten emitters and they appear to be more robust field emission electron sources. The smooth bare tungsten field emitters were irreversibly destroyed after only cycling them at increased vacuum pressures a couple of times, while the indium-coated emitters demonstrated the ability to be restored to their original performance by re-quenching them under ion-emitting conditions after being damaged.

Lifetime experiments demonstrated that the re-generable field emitters could sustain electron emission current for long periods of time and Fowler-Nordheim modeling of the emitter tip during voluntary shutdowns throughout the lifetime experiment yielded sharp emission sites on the order of 10's of nm. The sharp nano-

[§] where 'Unlimited' refers to the capability to re-generate the tip as long there is a sufficient metal supply available on the emitter tip.

structures successfully demonstrated electron emission at an emission current of about 3 μA for 1,750 hours from a single-needle that was quenched at an ion emission current of 20 μA . Emission current from the re-generable emitters stayed relatively constant for the long duration tests while the current degraded over time for both types of bare tungsten emitters.

Some of the emitter tips that were exposed to elevated vacuum chamber pressure demonstrated better electron emission performance after exposure. One possible explanation for this is that electron emission could have occurred from multiple emission sites simultaneously. During some of the experiments in the UHV chamber, when an emitter was used to obtain over 10 μA of electron emission current, multiple locations on the emitter tips could be observed through the optical microscope that were glowing blue. It is possible that the local electric field enhancement was very similar at numerous locations, so any of the locations that sustained damage due to elevated pressure conditions could have easily been replaced by other sharp locations. Another possibility is that ion sputtering at higher vacuum pressure could have actually sputtered away a few of the atoms on the field-emitting nano-structures, temporarily leaving an even sharper emitter.

VI. Conclusions

In order to determine performance characteristics of re-generable and tungsten emitters, lifetime experiments were performed using three types of field emitters; smooth bare tungsten, roughened bare tungsten, and the re-generable emitters. Single-needle tungsten field emitters were used for comparison rather than Spindt-type emitters to avoid complicated micro-fabrication that is required to build Spindt devices. Since Spindt-type emitters are made up of thin films of refractory metals, such as molybdenum or tungsten, the pure tungsten emitters should have had similar performance.

The main purpose of the comparison experiments was to determine if re-generable field emitters had similar emission characteristics to bare tungsten emitters and to determine what, if any, advantages re-generable emitters had over bare tungsten emitters that were approximately the same physical size and geometry. The experiments with the electrochemically DC etched smooth bare tungsten emitters demonstrated the least reliable operation and were the most prone to permanent damage after the fewest exposures to elevated vacuum pressure environments. The smooth bare tungsten emitters ceased to function after 1-2 cycles from 10^{-8} to 10^{-7} Torr. In addition, emission would cease after only a few minutes at increased pressure.

The experiments that were performed with the electrochemically DC and AC etched roughened bare tungsten emitters exhibited better performance than the smooth bare tungsten emitters. When exposed to increased vacuum pressure, the roughened emitters could sustain emission for up to 22 hours at pressure up to 10^{-5} Torr. Also, the roughened emitters survived 4-7 exposures to increased pressure before the emitters were permanently damaged. As mentioned, the smooth bare tungsten emitters lasted a maximum of 2 exposures. The increased lifetime was most likely due to the multiple nano-scale sharp ridges that were created during the AC electrochemical etch. Where the DC etched emitters were smooth and had a single sharp nano-structure, the AC etched emitters had multiple longitudinal nano-structures that were capable of field emission, which allowed the roughened bare tungsten emitters to last longer than smooth bare tungsten. However, emission current from the roughened bare tungsten emitters decreased over the duration of most experiments in UHV conditions, much like the Spindt-type arrays and carbon nanotube emitters in literature, indicating emitter tip degradation over time.

The re-generable emitters demonstrated the longest lasting performance at elevated pressure of the three emitters – even before re-generation. The re-generable emitters survived up to 45 hours at vacuum pressures up between 10^{-3} to 10^{-5} Torr. While the re-generable emitters could only be exposed to elevated pressure two times before emission could no longer be achieved at up to 4 kV of extraction voltage, they could then be re-generated into a like-new condition and subsequently operated for 100's of hours or longer. Therefore the re-generable emitters could potentially be cycled to increased pressure an almost unlimited number of times. Not only did the re-generable emitters prove to be the most reliable when exposed to unfavorable vacuum conditions, the emission current remained more stable (with less decrease) than the tungsten emitters throughout the duration of the lifetime experiments. Also, the experiments reported using Fowler-Nordheim modeling of re-generable emitters have estimated that the re-generable emitters can be used to create the same size nano-structures as state-of-the-art field emitters, 1's to 100's of nm.

Acknowledgements

Support from the Air Force Office of Scientific Research is gratefully acknowledged. The authors would also like to thank Marty Toth for his precision machining on all of the components that were used to perform the reported experiments.

References

- ¹ Marrese, C. M., "Compatibility of Field Emission Cathode and Electric Propulsion Technologies", *Doctoral Dissertation*, University of Michigan, 1999.
- ² Gamero-Castano, M., V. Hruby, P. Falkos, D. Carnahan, B. Ondrusek and D. Lorents, "Electron Field Emission from Carbon Nanotubes, and Its Relevance in Space Applications", *36th Joint Propulsion Conference*, AIAA-2000-3263, Huntsville, Alabama 2000.
- ³ Marrese, C. M., "A Review of Field Emission Cathode Technologies for Electric Propulsion Systems and Instruments", *IEEE*, 2000, 85-97.
- ⁴ Spindt, C. A., C. E. Holland, A. Rosengreen and I. Brodie, "Field-Emitter Arrays for Vacuum Microelectronics", *IEEE Transactions on Electron Devices*, 38, 10, 1991, 2355-2363.
- ⁵ Goldstein, J., Scanning Electron Microscopy and X-Ray Microanalysis, 3rd Ed. New York, NY: Springer, 2003.
- ⁶ Curtis, C. C. and K. C. Hsieh, "Spacecraft mass spectrometer ion source employing field emission cathodes", *Rev. Sci. Instrum.*, 57, 989, 1986,
- ⁷ Aplin, K. L., C. M. Collingwood and B. J. Kent, "Reliability tests of gated silicon field emitters for use in space", *Journal of Physics D: Applied Physics*, 37, 2004, 2009-2017.
- ⁸ Bonard, J.-M., C. Klinke, K. A. Dean and B. F. Coll, "Degradation and failure of carbon nanotube field emitters", *Physical Review B*, 67, 11, 2003,
- ⁹ Makela, J. M., R. L. Washeleski and L. B. King, "Re-generable Field Emission Cathodes Part I: Surface Morphology of In₂O₃ Emitter Apex ", *46th AIAA/ASME/SAE/ASEE Joint Propulsion Conference & Exhibit*, Nashville, TN 2010.
- ¹⁰ Park, Y. J., J. H. Choi, H. W. Lee, N. S. Lee, J. W. Kim, S. S. Hong, N. S. Park, J. E. Jung and J. M. Kim, "Fabrication of Spindt-type tungsten microtip field emitter arrays with optimized aluminum parting layers", *J. Vac. Sci. Technol. B* 18, 2, 2000,
- ¹¹ Makela, J. M. and L. B. King, "Re-generable Field Emission Cathodes for Low-Power Electric Propulsion", *43rd AIAA/ASME/SAE/ASEE Joint Propulsion Conference & Exhibit*, AIAA 2007-5171, Cincinnati, Ohio 2007.
- ¹² Makela, J. M., "Re-generable Field Emission Cathodes for Electric Propulsion", *Doctoral Dissertation*, Michigan Technological University, 2010.
- ¹³ Ekvall, I., E. Wahlstrom, D. Claesson, H. Olin and E. Olsson, "Preparation and characterization of electrochemically etched W tips for STM", *Meas. Sci. Technol.*, 10, 1999, 11-18.
- ¹⁴ Bell, A. E. and L. W. Swanson, "The Influence of Substrate Geometry on the Emission Properties of a Liquid Metal Ion Source", *Applied Physics A*, 41, 1986, 335-346.
- ¹⁵ Makela, J. M., R. L. Washeleski and L. B. King, "Re-generable Field Emission Cathode for Spacecraft Neutralization", *Journal of Propulsion and Power*, 25, 4, 2009,