Re-generable Field Emission Cathodes Part I: Surface Morphology of Emitter Apex

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This is the first part of a two-part paper that focuses on a field-emission cathode for use in Electric Propulsion (EP) that has the potential for very long lifetime due to its ability to be re-generated when the emitter tip become damaged. The field-emitting tips were formed by the application of an ion-extracting electric potential applied to a heated indium-coated tungsten needle, known as a liquid metal ion source (LMIS). The LMIS is then cooled, freezing in a solid nanotip at the apex. When the modified needle was then subjected to electron-extracting potentials stable and long-lived electron emission was observed. The focus of this investigation was to operate and quench a LMIS at ion emission currents from 2 to 25 μA to acquire micrographs of the surface morphology as a function of the ion emission current at quench. The LMISs were also operated at selected ion emission currents for 1's to 10's of seconds between quenching to observe the temporal change in emitter tip surface morphology as a function of ion emission current. Micrographs of the quenched emitter tips yielded Taylor-cone-shaped structures. The quenched emitters exhibited multiple nano-protrusions on the surface of the micro-scale Taylor cone, which were capable of electron field emission.

I. Introduction

HISTORICALLY, liquid metal ion sources (LMISs) have found extensive use as ion sources of high brightness in focused ion beam materials processing applications[1] and, more recently, as electric propulsion thrusters via FEEP technology.[2-4] In an LMIS or FEEP thruster, an intense electric field is created near the surface of a low melting-temperature liquid metal, such as indium, by a downstream electrode. A balance between the liquid surface tension and electrostatic forces cause a structure known as a Taylor cone to form in the liquid.[5] Because the Taylor cone has a very sharp tip, geometric enhancement of the local electric field at the cone tip is sufficient to extract metal ions directly from the liquid. The ions emerge from a very narrow (few nanometer diameter) liquid jet at the cone apex and are subsequently accelerated by the electric field to either produce thrust (FEEP) or for materials processing applications (LMIS). Other applications and areas of interest for the use of focused ion beams include lithography, semiconductor doping, sample preparation for TEM imaging, circuit repair, scanning ion microscopy, and scanning ion mass spectroscopy.[6]

For low-power electric propulsion applications, e.g. FEEP systems, an electron source is a necessary thruster component to maintain spacecraft neutrality since an operating thruster will cause a global charge imbalance on a spacecraft. Typical electron sources, or cathodes, that are used with low-power thrusters are field emission cathodes. Field emission cathodes use nano-scale sharpened electrodes with locally enhanced electric fields to cause electrons to escape from the surface of the electrode into vacuum via a quantum tunneling effect known as Fowler-Nordheim emission. The local electric field 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. Many types of electron field emitters have been used in the past, with the most popular being the Spindt-type array[7] and, more recently, carbon nanotube field emission arrays.[8] The only drawback to field emission cathodes is the limited lifetime associated with the devices. The nano- or micro-scale features are fragile and when the features become damaged, the electron source loses functionality. Researchers have found some ways to minimize damage to the emitters[9] and they have also found more robust, longer-life, emitter materials.[8, 10-12] However, all electron field emitters become damaged over time[13, 14] – it's just a matter of how much time it will take.

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In 2007, Makela and King proposed and demonstrated a technique for re-generating solid-metal field-emitter tips using a liquid-metal ion source.[15-17] The LMIS was used to construct nano-scale metal structures intended for use as electron field-emission neutralizers for space applications. The feasibility of creating field emitting tips by quenching the ion emitting LMIS at emission currents ranging from 1 to 25 μA was demonstrated. It was shown that sharp nano-structures could be regenerated as long as there was a sufficient supply of indium to obtain ion emission. It was also found that the electron I-V characteristics of a field emitter could be altered by changing the ion emission current during the quench. The results of those experiments showed that as ion emission current during quenching was increased, the subsequent electron emission performance increased – inferring that the resulting solid emitter tip radii decreased. Applying the Fowler-Nordheim model to the electron I-V data yielded tip radii ranging from 230 to 80 nm at quench currents of 1 to 25 μA, respectively.[16-18]

II. Goal of Study

The primary goal of the research reported in this document was to examine the nanostructures formed during quench of an operational LMIS using electron microscopy and to understand how the morphology of these structures depends on the ion emission parameters during the generation process. The research reported in this paper focuses on investigating the geometry of indium emitter tips that were formed by quenching an operating LMIS. Multiple tests were performed under a range of ion emission current conditions and then the tips were observed using a Field Emission Scanning Electron Microscope and the results were compared with Fowler-Nordheim models of data collected from the experiments. For each experiment the extraction electrode voltage was varied to investigate the emitter tip shape at ion emission currents ranging from 2 to 25μA.

III. Custom FE-SEM apparatus

The emitter fixture that was used for the FE-SEM experiments is shown in Figure 1. As shown, some modifications were made to the FE-SEM load-lock chamber that allowed a liquid metal ion source (LMIS) to be placed in the specimen chamber of the FE-SEM to perform emitter re-generation experiments *in situ* within the microscope. The custom fixture was equipped with electrical connections to operate the resistive heater and extraction electrode that are necessary to operate the LMIS. The electrical connections were made by three stainless steel discs that were connected to the heater and extraction electrode and were located underneath the top Teflon surface, shown in Figure 1. The three discs were used to mate with the internally mounted electrical interface. Implementing the custom fixture and electrical connections allowed the dual ion/electron source to be operated in the specimen chamber of the FE-SEM.

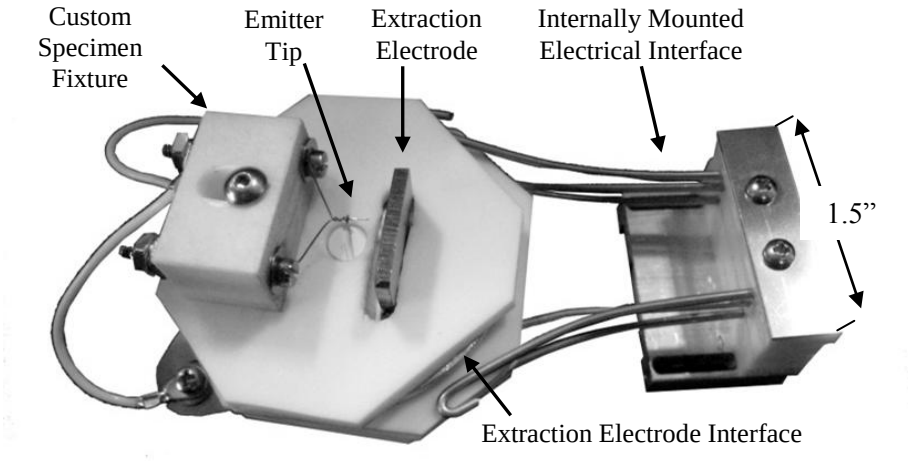

Figure 1. Dual electron/ion source apparatus and the custom electrical interface mounted inside the Field Emission Scanning Electron Microscope.

The chamber was evacuated using a series of three ion pumps and vacuum pressure of 10^{-7} Torr was maintained throughout testing. An electrical schematic of the completed re-generable emitter apparatus is shown in

Figure 2. For vacuum compatibility, the materials that were used to build the custom LMIS and support fixture included Teflon, stainless steel, and tungsten. Gap spacing between the emitter tip and the extraction electrode was ≤ 0.5 mm for all of the experiments.

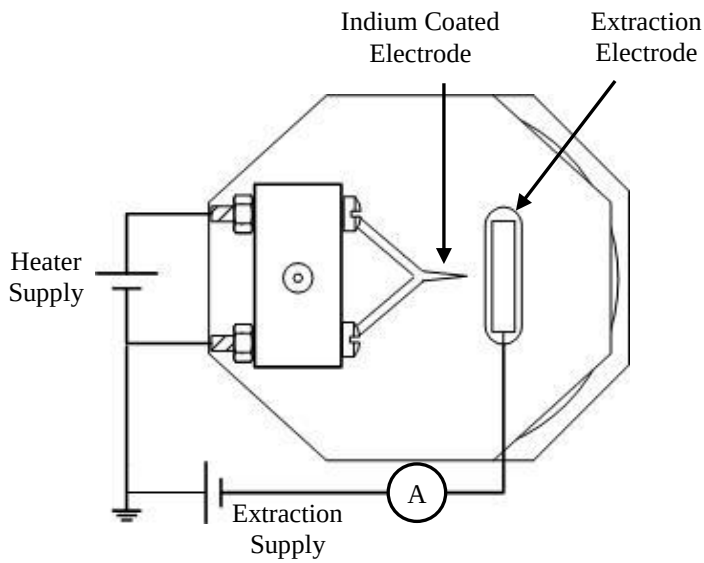

Figure 2. Electrical schematic of the FE-SEM specimen fixture showing the heater supply, the extraction supply, and the ammeter placement.

The internal electrical interface was installed permanently inside of the FE-SEM specimen chamber. Electrical connections were made by inserting the custom specimen fixture so that the stainless steel electrodes that were connected to the heater and extraction electrode on the specimen fixture were made continuous with the internally mounted electrical interface by surface contact.

To achieve ion emission, the resistive emitter heater, shown previously in

Figure 2, was supplied with 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 extraction supply voltage was then increased until ion emission was established. For each set of experiments the extraction supply was increased until the desired ion emission current was reached and then the extraction electrode voltage was held constant while a pre-determined time was allowed to elapse, t_e. Leaving the extraction voltage constant, the heater power was turned off to quench the operating ion source. After the LMIS was allowed to cool for 30 seconds, the extraction supply was also turned off. Once the heater and extraction power supplies were off, the electron optics on the FE-SEM were engaged and micrographs of the emitter tip were acquired. After imaging the emitter tip, the electron optics were turned off and an electron I-V sweep was performed to apply to the Fowler-Nordheim model for tip radii estimations. The process of tip re-generation with subsequent imaging and electron I-V acquisition was repeated multiple times at a range of ion emission currents from 2 to 40 μA and a range of t_e from 10 to 240 seconds.

Between successive experiments the emitter tip surface morphology was 'reset' to eliminate any nano- or micro-structure on the surface. To 'reset' a smooth surface, the extraction power supply was current-limited at 100 μA while increasing the extraction voltage to about 7 kV. An emission current of 100 μA was sufficient to melt the sharp tips and the extraction voltage was high enough to cause arcing to destroy any locally sharp points on the apex.

IV. Experimental Results and Discussion

Two sets of experiments were performed within the FE-SEM and are reported in Section IV.A and IV.B. Section IV.A describes the temporal evolution of a re-generable emitter tip. Section IV.B is comprised of micrographs of quenched emitters and Fowler-Nordheim analyses from the quenched emitters at ion emission

quench currents ranging from 2 to 20 μA. The data presented in Section IV.B were then compared with data that were reported from similar experiments in an ultra high vacuum facility.[16]

A. Temporal Nano-structure Formation

It is well known that ions are emitted from a single jet-like protrusion when operating a liquid metal ion source.[19, 20] Therefore, prior to this investigation it was thought that a single jet-like protrusion would be solidified upon quenching an operating ion source. To investigate the surface morphology, the re-generable source was operated at ion emission currents of 10, 20, and 30 μA for 10-second intervals and imaged at each interval. The first experiment was performed at 10 μA of ion emission current, quenching the source every 10 seconds to show the evolution of the emitter tip after seven consecutive quenches at a constant ion emission current, as shown in Figure 3. For the 10-μA quenches, successive quenches created surface modification after about 40 seconds of ion emission.

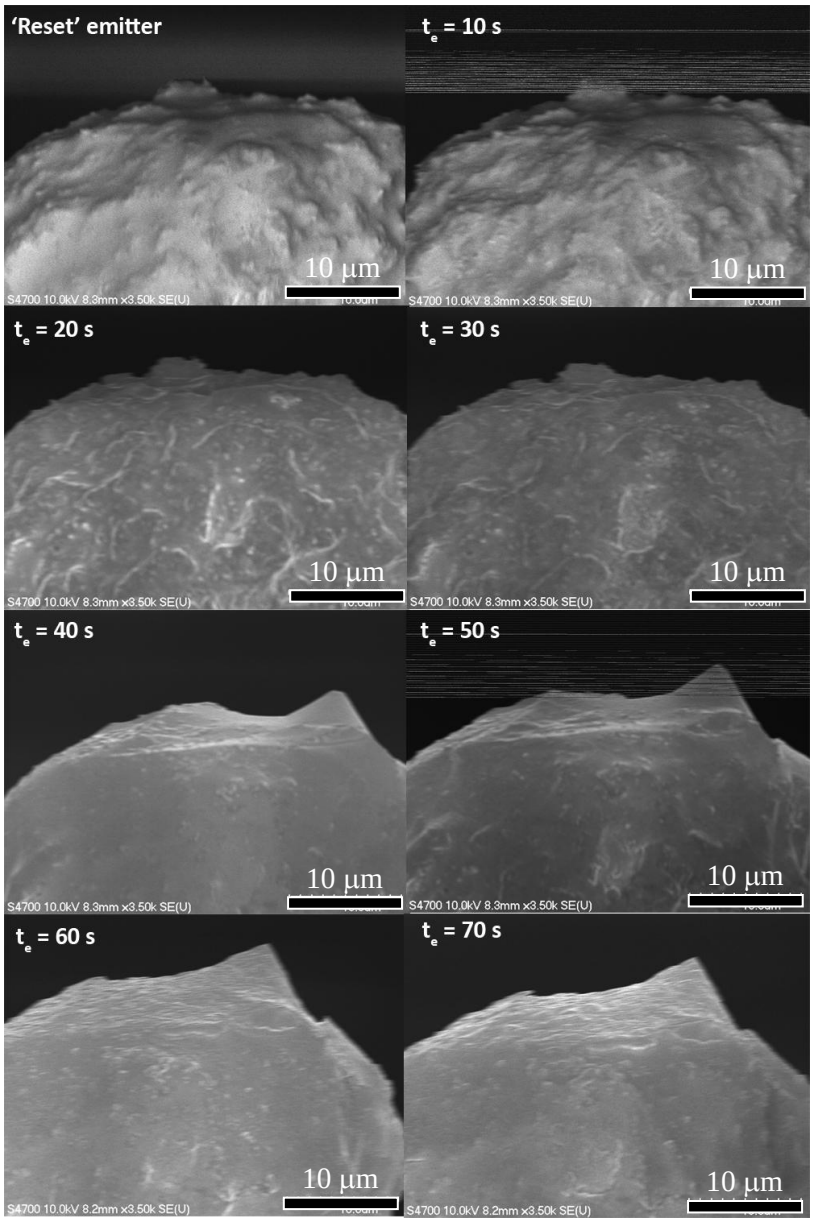

Figure 3. FE-SEM micrographs of the emitter tip taken after consecutive quenches at ion emission current of approximately 10 μA.

As shown in the micrograph, slight surface modification occurred after the first 10-second-quench at 10 μA. After the next quench for 10 seconds the surface roughness appears to get more defined and not much changes between the second and third quench. After the fourth quench the emitter tip appears to have grown a Taylor cone structure. After the fifth quench of 10 seconds at 10 μA, the Taylor cone becomes more pronounced. After the sixth and seventh quenches, surface texture starts to become visible on the surface of the Taylor cone.

The same emitter was then reset and used to observe nano-structure formation at higher ion quenching currents. The experiment was performed by operating the re-generable source at an ion emission current of 20 μA with about 3 W of heater power for a quantity of seven 10-second intervals. After each time the emitter was quenched a micrograph was acquired, as shown in Figure 4. Just as with the 10-μA experiment the images are intended to show the temporal evolution of the emitter tip after consecutive quenches at a constant ion emission current of 20 μA. After the first quench, at t_e = 10 s, a large cone formed at the center of the emitter apex. After 10 additional seconds of operation the cone appeared to retract and some nano-structures began to form. At an elapsed time of 30 s a very well structured Taylor cone formed with some nano-structure along the surface of the apex. Additional ion quenches didn't generate many more nano-structure beyond the first 30 s.

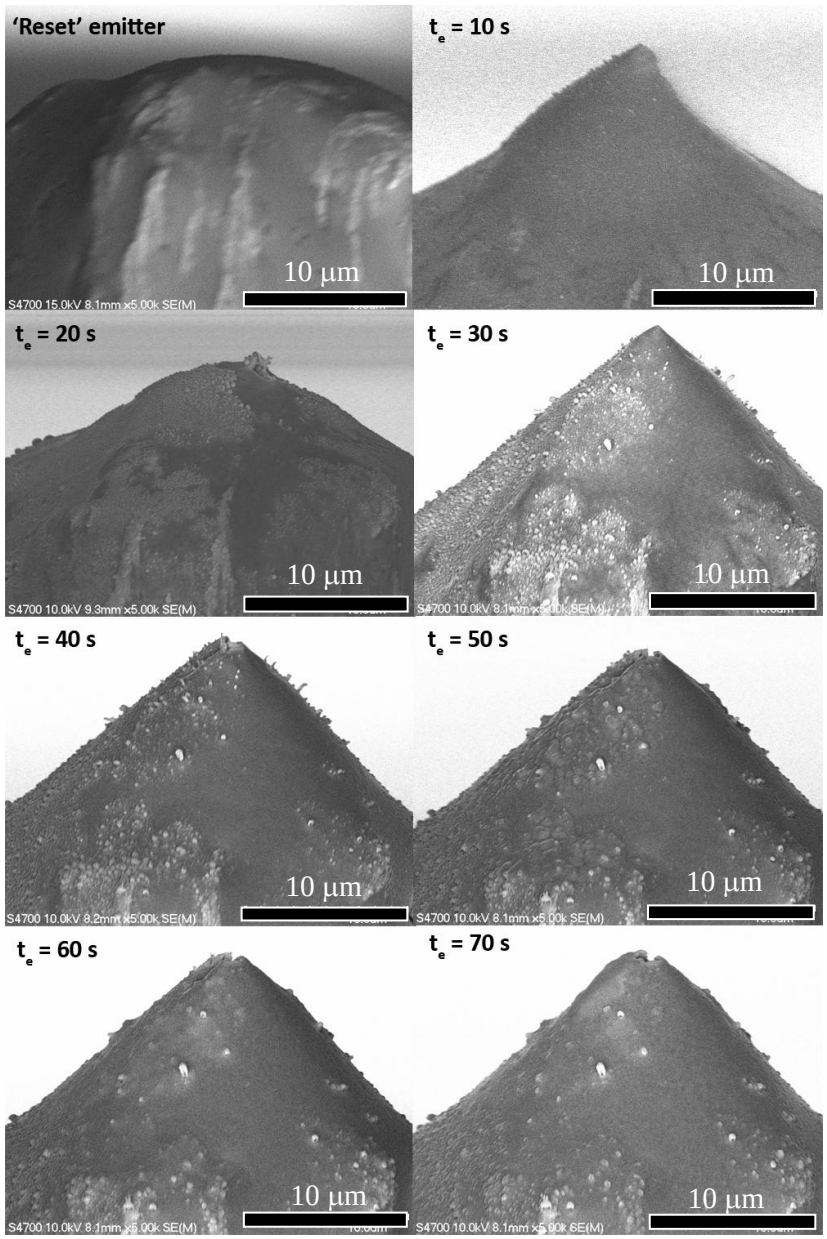

Figure 4. FE-SEM micrographs of the emitter tip taken after consecutive quenches at ion emission current of approximately 20 μA.

The micrographs show an interesting depression at the apex that resembles a "micro-volcano" that formed after about 40 seconds of operation. It is possible the surface of the Taylor cone formed a thin oxide layer, which could be responsible for holding the shape of the emitter between successive quenches. Indium tends to form indium oxide, In_2O_3, even when in a vacuum environment. Indium oxide melts at 1910°C so it could be possible that during subsequent quenches the emission current was sustained at the apex by liquid indium (T_{melt} = 156.6 °C) beneath the oxide layer that was able to break through the layer, resulting in the "micro-volcano" structure due to depletion of indium. The emitter tip most certainly had to be at a greater temperature than the melting temperature of indium in order to sustain ion emission, which was the intent of heating the source with about 3 W during testing. Off-the-shelf LMISs used for Focused Ion Beams are similar to the emitters used for the testing reported here and typically use about the same amount of heater power. Whether indium or In_2O_3 the "micro-volcano" structure is not completely understood at this time.

After seven quenches, totaling 70 seconds of ion emission, the same re-generable source was again re-set as described in Section III. The re-generable source was operated at 30 μA of ion emission current and then quenched

after 10 s of operation. The emitter tip was imaged and operated at 30 μA of ion emission current for six additional quenches at 10 second intervals, as shown in Figure 5.

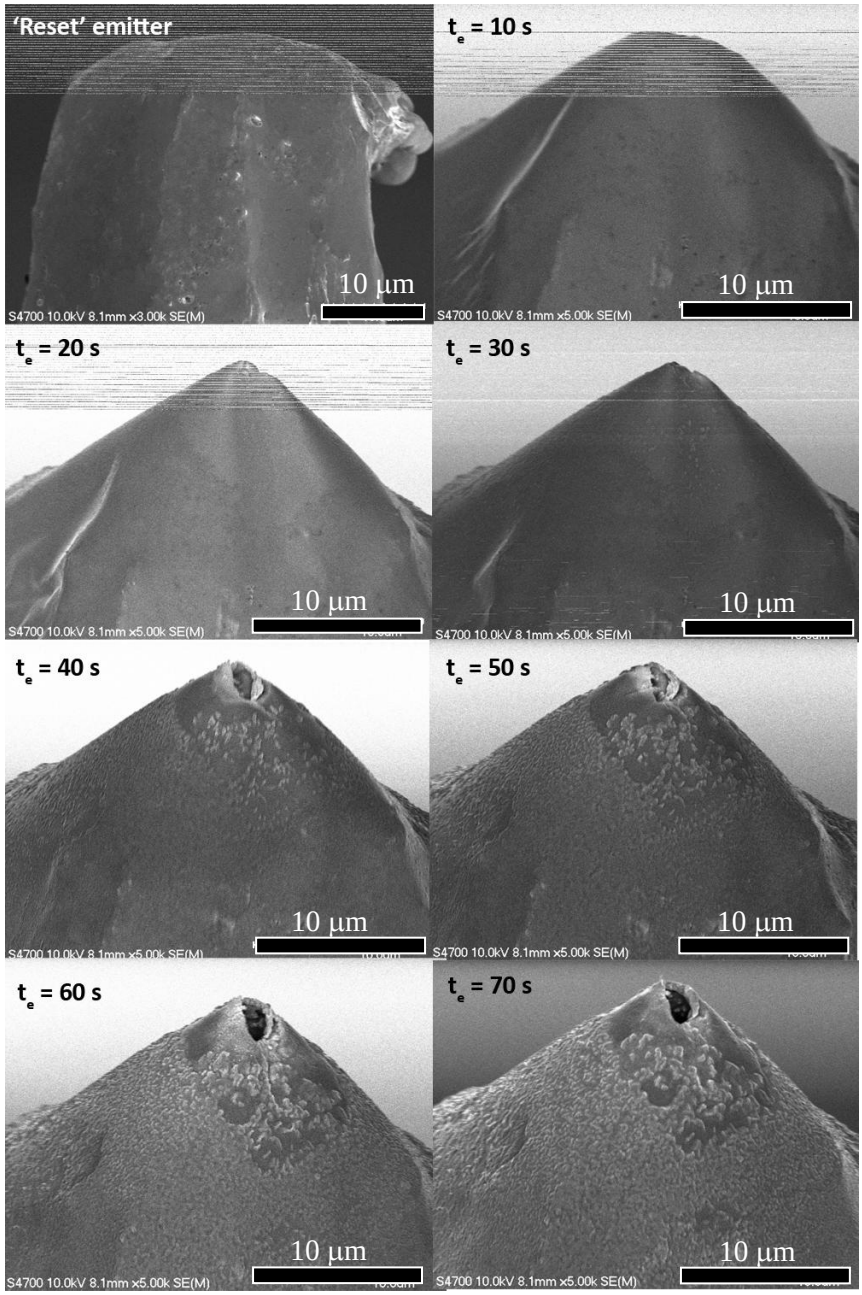

Figure 5. FE-SEM micrographs of the emitter tip taken after consecutive quenches at ion emission current of approximately 30 μA.

The apex of the emitter began to form a cone after the first 10-second-quench. The cone became more defined after 20 seconds of operation and some nano-structure began to form along the tip's surface. It wasn't until after the third 10-second-quench that the nano-structure really started to become defined, as shown in the micrographs. Also, the "micro-volcano" structure appeared after the fourth quench, which was at the same point as in the 20-μA series when the structure formed.

To determine if the micrographs showed the evolution of the emitter tip or if the emitter tip relaxed after each time it was heated (between pictures in a given series of images) and formed a new structure during each 10-second emission iteration an experiment was performed to determine how much the tip would 'relax' during a

heating cycle with no ion emission. This experiment consisted of 'resetting' an emitter tip, operating the emitter at 20 μA of ion emission current for 20 seconds, and then quenching the emitter to form a Taylor cone. The Taylor cone was imaged with the FE-SEM and then heated in the absence of an electric field (no ion current) to observe if the emitter tip relaxed, as shown in Figure 6.

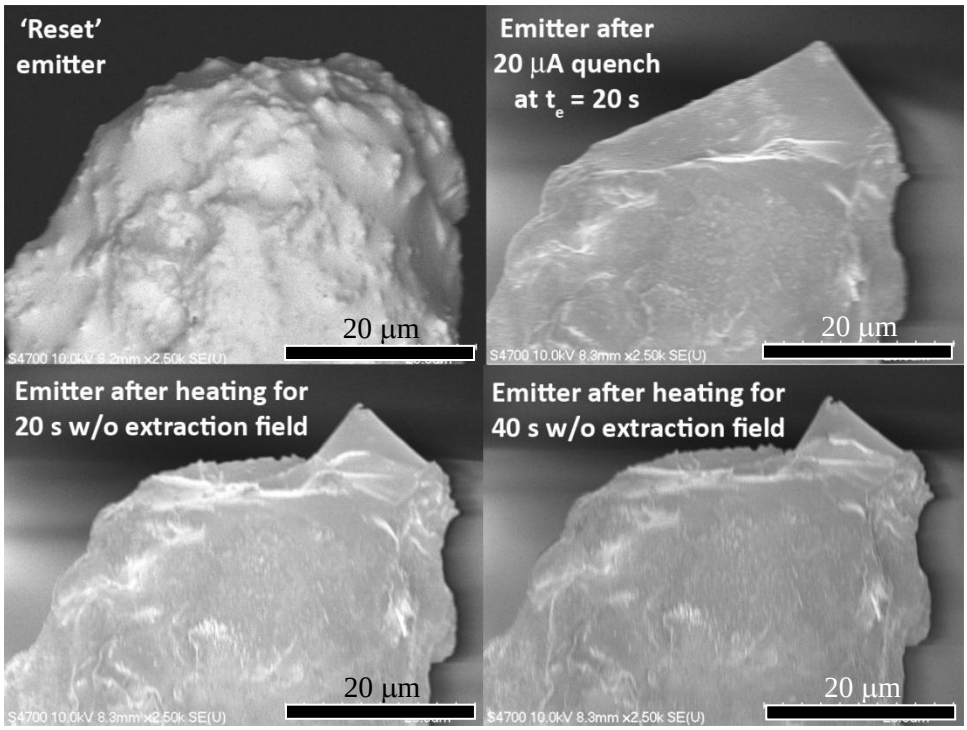

Figure 6. Micrographs of a 'reset' emitter, after operating the emitter for 20 seconds at 20 μA, and then two micrographs acquired after heating the emitter for 20-second intervals in the absence of an electric field, showing that the emitter tip doesn't completely relax between consecutive quenching experiments.

The same emitter was then operated at 20 μA of ion emission current (*sans* resetting) by heating the emitter and increasing the extraction voltage to establish emission. Ion emission was adjusted to 20 μA for 20 seconds and then quenched. After quenching, the emitter was imaged and heated for 20 seconds in the absence of an electric field. Following the heating experiment, the emitter was imaged and heated again for an additional 20 seconds, as shown in Figure 7.

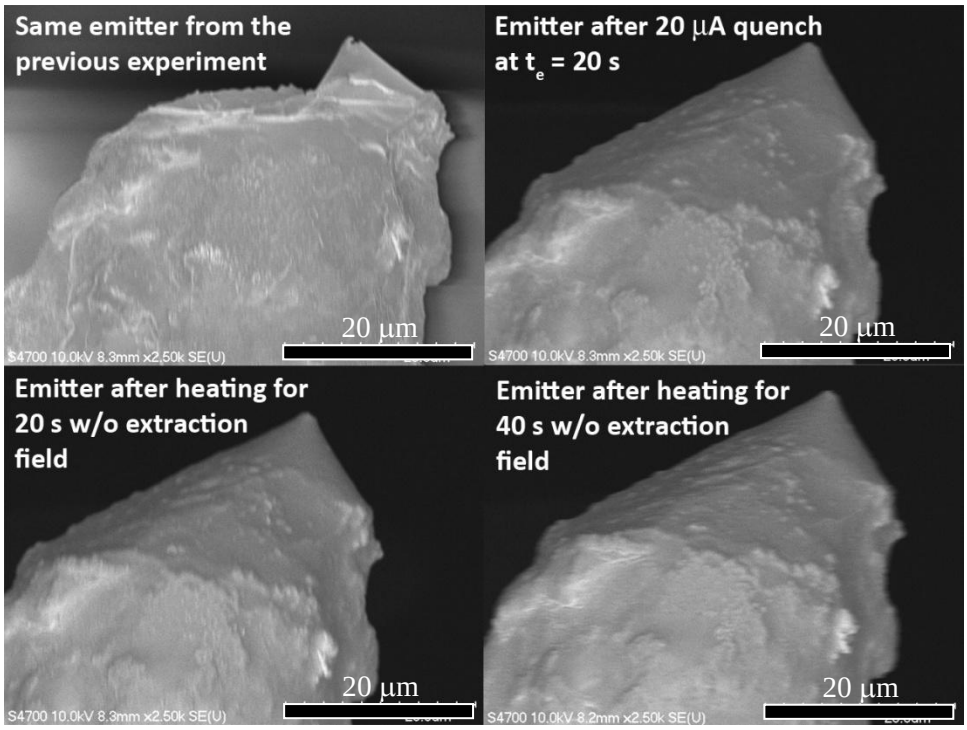

Figure 7. Micrographs of the same emitter shown in Figure 6 and then after operating the emitter for 20 seconds at 20 μA, followed by two micrographs acquired after heating the emitter for 20 second intervals in the absence of an electric field, showing that the emitter tip doesn't relax between consecutive quenching experiments.

It is impossible to say with certainty that the surface morphology remains the same between subsequent thermal cycles but the micrographs show that the emitter tip surface doesn't completely relax during each test. Since the emitter tip didn't completely relax between successive tests, the images in Figure 3 through Figure 5 likely show the change in surface morphology as a result of the cumulative ion emission time.

B. Nano-structure Formation with Fowler-Nordheim Modeling

The purpose of this experiment was to use the FE-SEM to visually verify the trends that were observed via Fowler-Nordheim modeling of data taken in an earlier study within a UHV chamber[16] In these earlier tests, the UHV chamber was equipped with an optical microscope that only had 90X magnification, which wasn't high enough magnification to resolve the nano- and micro-structure of quenched emitters. Obtaining higher resolution micrographs motivated this series of experiments.

The re-generable emitters were quenched at multiple ion emission currents inside the FE-SEM. The FE-SEM was then used to image the nano-structure formation after quenching. Also, electron emission I-V sweeps were performed after each quench to estimate the emitter tip radii using the Fowler-Nordheim model. The emitter tip estimations were compared with the FE-SEM micrographs. The ion quenching currents that were chosen for the experiment ranged from 2 to 20 μA, operating each for 2 minutes exactly as done by Makela et al.[16] After each quenched emitter was imaged and an electron I-V sweep was acquired the emitter tip was then 'reset' using the same procedure described previously of exposing the emitter tip to destructive electron emission conditions to smooth out and destroy surface features. The quenching experiments were performed in a randomized order but will be presented from lowest-to-highest ion quenching current. The actual order of ion emission current before quenching was 20, 10, 5, 15, 20, 6, 16, 3, 10, and then 2 μA. After showing the individual results, a summary will be provided with all of the compiled data, including data from previous work in the UHV chamber.[16]

For each experiment the re-generable emitter was first exposed to electron 'reset' conditions. For the 1st reported experiment, the emitter was operated at 2 μA of ion emission current (at 3.3 kV) for 2 minutes and quenched. The image in Figure 8 shows a post-quench micrograph at 9000X, the highest resolution that was possible to obtain during testing. As shown, a cone-type structure formed with nano-scale features on the surface. Also, the volcano-type structure that was observed in the previous section was present in this test.

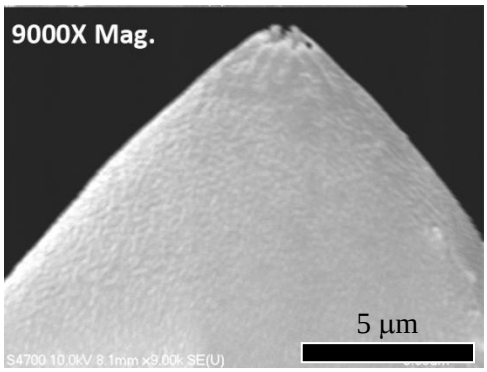

Figure 8. Micrograph showing a re-generable emitter tip after a 2 μA quench for 2 minutes at magnification of 9000X.

After quenching and imaging the emitter tip, an electron I-V sweep was performed on the solidified emitter by increasing the extraction voltage up to 2.9 kV at 50 volt increments. A Fowler-Nordheim plot was created from the sweep data, as shown in Figure 9, with an R^2 of 0.86 for the linear curve fit. Applying the Fowler-Nordheim model to the data resulted in an emitter tip radius estimate of 7.0 ± 1.4 nm.

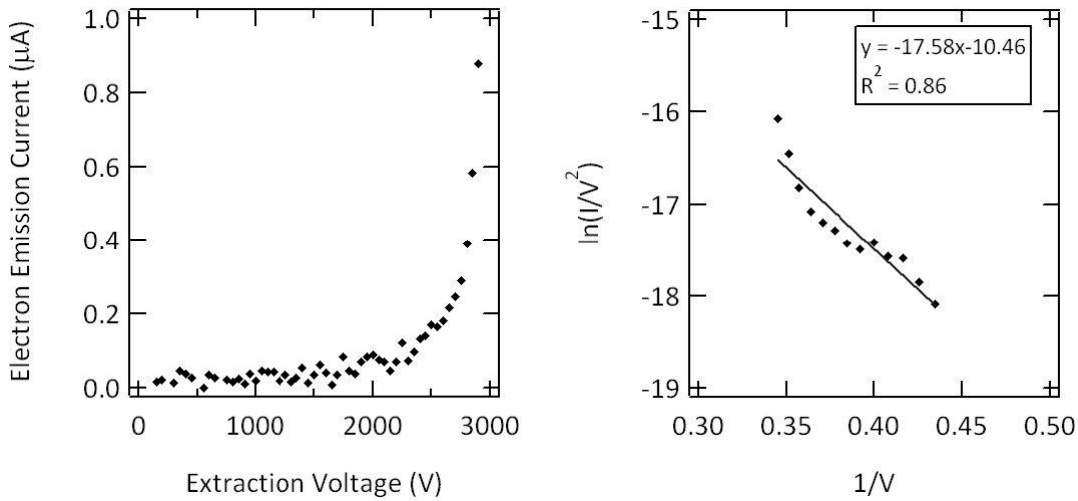

Figure 9. Fowler-Nordheim plot from a re-generable emitter quenched after 2 minutes at 2 μA, yielding a tip radius estimate of 7.0 ± 1.4 nm.

After generating an emitter, imaging it, and acquiring Fowler-Nordheim data, the emitter was 'reset', re-generated, and the process was repeated. For each new experiment, the ion current at quench was adjusted to a value between 2 and 20 μA and then the quenched nano-structures were imaged using the FE-SEM and subjected to an electron I-V analysis. Table 1 shows the test conditions that were explored and includes the ion current before quench and the extraction voltage at quench.

Table 1. Settings for tip re-generation experiments. The ion emission current and extraction voltage at quench are presented, as well as the chronological order that the data points were acquired and the extraction voltage required for about 1 μA of electron emission current.

Ion Current at Quench (μA)	Extraction Voltage at Quench (kV)	Chronological Order of Data
2	3.3	10
3	3.4	8
5	3.8	3
6	3.2	6
10	3.5	2
10	3.4	9
15	3.9	4
16	3.4	7
20	3.7	1
20	3.8	5

The FE-SEM micrographs from representative experiments are shown in Figure 10 and Figure 11. Each quenched emitter tip exhibited nano-structure formation on the surface. Many of the emitter tips also formed the "micro-volcano" structure. The complete set of micrographs are available in Makela.[21]

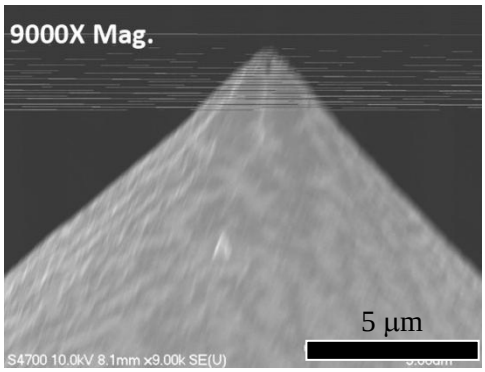

Figure 10. Micrograph showing the emitter tip nano-structure after a 10 μA quench for 2 minutes.

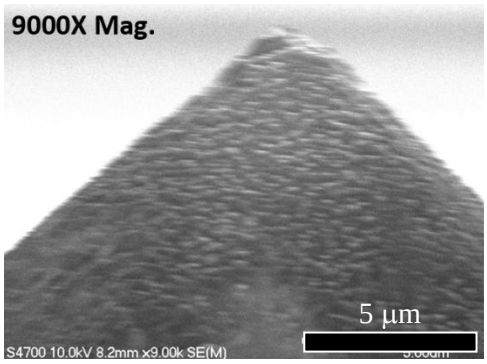

Figure 11. Micrograph showing the emitter tip nano-structure after a 20 μA quench for 2 minutes.

As shown, the emitter tips are on a micro-scale with nano-scale features that formed on the surface of the emitters. Although the exact size of the nano-features can't be distinguished, electron I-V sweeps were acquired

from each quenched emitter tip so that Fowler-Nordheim estimations could be made. All of the electron I-V data and the Fowler-Nordheim plots that were acquired in the FE-SEM are summarized in Table 1 and the data are plotted in Figure 12. Also reported in the table is the R^2 value from each of the Fowler-Nordheim analyses. Included on the plot is the data taken in the UHV chamber.[16] As shown, within the error bars there isn't a discernable trend in the emitter tip radius as the ion current is varied.

Table 2. Emitter tip radius estimates from Fowler-Nordheim analyses of re-generated emitter tips at currents ranging from 2 to 20 μA.

Ion Current at Quench	Emitter Tip Radius Estimate	R^2 value from F-N Plot
μA	nm	
2	7.0 ± 1.4	0.86
3	28.9 ± 5.8	0.96
5	22.6 ± 4.5	0.90
6	9.0 ± 1.8	0.91
10	5.5 ± 1.1	0.89
10	14.1 ± 2.8	0.79
15	29.6 ± 5.9	0.80
16	9.1 ± 1.8	0.88
20	27.3 ± 5.5	0.79
20	20.4 ± 4.1	0.77

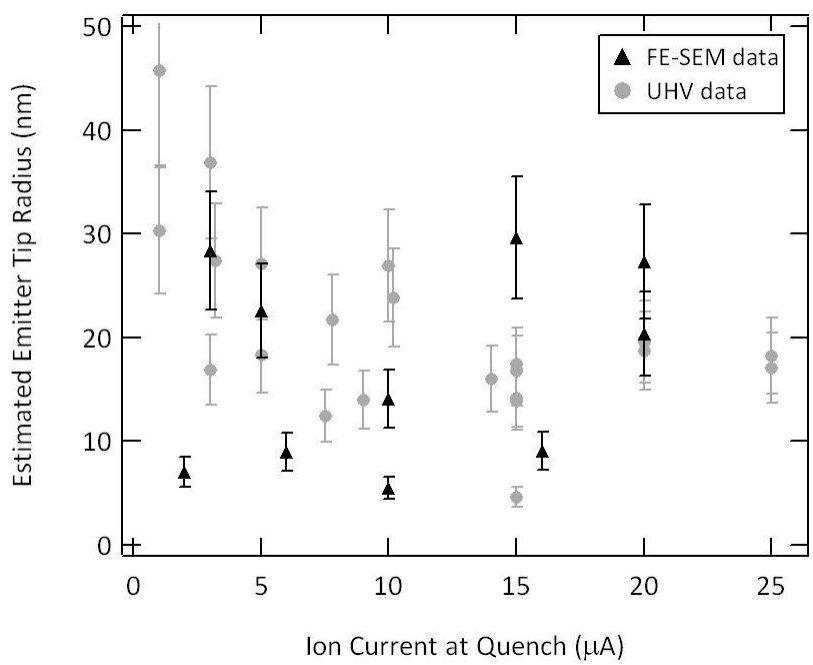

Figure 12. Data from experiments performed inside the FE-SEM and experiments performed in the UHV chamber that were reported by Makela, Washeleski, and King[2] showing estimated emitter tip radii, using Fowler-Nordheim modeling, at ion currents before quenching of 1 to 25 μA.

Looking at the FE-SEM micrographs shown previously, it was not possible to make accurate emitter tip radii estimations visually. Unfortunately, the resolution of the micrographs was limited due to vibration in the fixture, since the LMIS was cantilevered off of a Teflon block. When the Fowler-Nordheim data that were acquired

in the FE-SEM were combined with the data taken in the UHV chamber the plot in Figure 12 was generated. All of the data points that were obtained by Fowler-Nordheim modeling of electron I-V curves taken from quenched field emitters were in the same range. As shown, there is up to 25 nm of scatter in the data but all of the emitter tip radii estimations were between 5 and 50 nm.

Investigation of the surface structure of a quenched ion source using a Field Emission Scanning Electron Microscope resulted in the observation of a Taylor cone shaped structure, which was expected. However, each cone had multiple nano-structures that were solidified on the surface of the emitter tip. The micrographs revealed that the nano-structures were evenly spaced and approximately the same size all over the surface. Although electron emission was most likely occurring at the apex of the emitter due to the apex being in closest proximity to the extraction electrode, any of the nano-structures are capable of emission if the sharpest structures at the apex become incapable of emission.

The nano-structures were formed from quenching a liquid metal ion source at ion emission currents ranging from 2 to 30 μA. It was shown that a well-defined Taylor cone substructure forms after 20 to 30 seconds of ion emission and that the number of nano-structures that are formed on the surface of the Taylor cone tends to reach a maximum after 30 to 40 seconds of ion emission. The exceptions were two of the quenches, for unknown reasons a 5 and 15 μA quench didn't produce a Taylor cone substructure or multiple nano-structures, however, they still had some sharp nano-structure. Another interesting observation was that the Taylor cones didn't completely melt by adding heat in the absence of an electric field. While the temperature of the indium must have been sufficient to liquefy some of the indium, since ion emission could be established and the bulk shape of the emitter could be deformed, it is possible that an indium oxide coating on the surface of the emitter could have been responsible for maintaining the shape of the emitter when heated without an applied electric field. As discussed in Section IV.A, indium oxide, In_2O_3, has a much higher melting temperature than indium so the oxide layer could have formed a solid 'crust' on the emitter surface. The Fowler-Nordheim model approximations reported in this paper had comparable tip radii estimations to those reported previously by Makela et al.[16] Visually inspecting the nano-structures with the FE-SEM didn't provide sufficient resolution to accurately determine the emitter tip radii, however, Fowler-Nordheim model radii estimations ranged from about 5 to 50 nm.

V. Conclusions

The main purpose of the FE-SEM experiments was to observe what happened to the surface morphology after quenching a liquid metal ion source and to compare the results with Fowler-Nordheim modeling. From literature, it was expected that a single jet-like protrusion would form at the tip of a Taylor cone. It was also expected that the geometry of the single protrusion would change depending on the ion emission current that was being emitted upon quenching. A Taylor cone structure did form, however a jet-like protrusion could not be detected from the micrographs. An interesting and un-anticipated observation from the micrographs revealed multiple nano-structures of similar geometry covering the apex of the emission electrode. As the quenching process was repeated, the protrusions that were formed became more and more defined and between subsequent quenches the overall structure of the Taylor cones maintained their shape, even when tested solely with heat in the absence of an electric field.

Formation of multiple protrusions from a quenched liquid metal ion source was a phenomenon that had never been observed but is very advantageous for re-generable cathodes. Since all of the nano-structures had very similar electric field enhancement, emission could have occurred from multiple nano-structures simultaneously. The multiple nano-structures that were formed on the re-generable field emitters could even act as a field emitting array, like Spindt-type arrays and carbon nanotube mesh.

Another interesting feature was also present in many of the micrographs that were taken after quenching an operating liquid metal ion source multiple times. A hollow depression that looked like a "micro-volcano" formed at the emitter tip apex after subsequent 10-second quenches and appeared to form after the fourth 10-second quench in most cases. It is not clearly understood at this time but it is possible that a thin oxide layer is present on the surface of the emitter, causing the emitter tip to retain its shape during repeated quenching while the emission is sustained by depleting liquid indium from the apex.

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