Instrument for Characterization of Field Emission Properties of Nanostructured Surfaces

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In this paper, an instrument for the characterization of field emission properties of nanostructured surfaces is described. First presented is an introduction to the theory of field emission followed by a short derivation of the key equations formulated by Fowler and Nordheim. The system is then described followed by an analysis of the system’s performance and some example results.
Acknowledgements

I would like to thank Professor Lambeth for his guidance, his bottomless well of knowledge and his patience. I’ve learned a lot from him and I hope to learn much more. I’d like to thank Chando Park for all his help in understanding this material. His advice as well as that of Bo Li and Suresh Suranthem made this work possible. I’d also like to thank Lin Wang, Nancy Dean, Bo Li, Chando Park, my parents and my girlfriend Ana Carter for their support.

Gilad Kusne
Section 1 Introduction

In 1956, field-induced electron emission placed last when compared to the technical significance of thermionic emission, photoelectric emission and secondary emission\(^1\). Extremely high fields were needed to induce useful emission current densities, making field-induced emission the least feasible form of electron emission for emerging technologies. At the time it was understood that topological nanostructures on an emitting surface could solve this problem by intensifying and focusing the localized field, thus reducing the field strength one needed to apply to induce emission currents - experimental results showed local field amplifications by as much as two or three orders of magnitude\(^2\). However, controlling the shape and ordering of nanostructures was still an immature art. The advent of the Scanning Electron Microscope, the Atomic Force Microscope, and the Scanning Tunneling Microscope sped up the development of this art and promoted the technological feasibility of field-induced electron emission. Today field-induced electron emission is used in technologies such as field emission displays\(^3\), field emission microscopy\(^4\), and pressure sensors\(^5\).

One of the primary champions of field-induced electron emission is the display industry. Much study has gone into a technology that promises to rival the top three technologies of our time – the Cathode Ray Tube (CRT), the Liquid Crystal Display (LCD) and the Plasma Display (PDP). A Field Emission Display is expected to offer the superior luminescence, fuller color, faster response time and wider viewing angle.

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of the CRT in a package as thin as a PDP and as light as an LCD\(^1\). With the proper materials, field-induced emission may overtake these display technologies. The instrument presented here allows characterization and categorization of materials for their potential impact as emitters in such an industry.

In particular the instrument was constructed to allow characterization of a novel nanostructured carbon films. The film’s nanostructure is obtained through the use of self-assembling block copolymers\(^2\). This chemical process can be used to manufacture nanostructured carbon films on a large scale and has the potential to replace the current methods of carbon film production: CVD, laser ablation, and arc welding. Nanostructured carbon films made by variations in the block copolymer processing technique will be tested for field-emission qualities to determine the optimal processing parameters for generating high field amplification field-emission films.

\(^1\) Mann C. (Nov. 2004). Nanotech On Display. *MIT: TechnologyReview.com*

Section 2 Theory

2.1 Fowler-Nordheim Equation Explained

The science of field emission began in 1928, when Fowler and Nordheim presented the first quantum-mechanical model for describing field induced electron emission from a metallic surface; a model still in use today. In deriving their model, Fowler and Nordheim first assumed that the conduction electrons in the emitting metal are describable as a free-flowing 'electron cloud' - following Fermi-Dirac statistics - and are bound to the metal by an energy barrier at the surface. Under the influence of a field, these conduction electrons can be induced to tunnel through the barrier into vacuum, producing a field-induced electron emission from the metal surface. Fowler and Nordheim further assumed that the surface barrier can be approximated with a one dimensional energy function without losing significant accuracy. (The equations presented here assume a Fermi-Dirac metal emitting surface. Formulas for field emission from a semiconductor, specifically from a semiconductor conduction or valence band, can be found in Stratton’s “Theory of Field Emission from Semiconductors”).

\[ V(x) = E_{\text{vac}} - eFx \]

Figure 1: Surface energy barrier at metal-vacuum interface.

---

Assuming that the emitting metal surface lies perpendicular to the x-axis as sketched in Figure 1, the electron emission current density from the surface of the metal is described by the equation:

$$J_x = \int D(\varepsilon_x)N_x(\varepsilon_x)d\varepsilon_x \quad [1]$$

Where $N_x(\varepsilon_x)d\varepsilon_x$ is the density of electrons within the metal with kinetic energy $\varepsilon_x$ in the x-direction within a kinetic energy range $d\varepsilon_x$, and $D(\varepsilon_x)$ is the probability of an electron with kinetic energy $\varepsilon_x$ tunneling through the surface barrier.

$N_x(\varepsilon_x)d\varepsilon_x$ is determined by solving:

$$N_x(\varepsilon_x)d\varepsilon_x = \iint_{k_x,k_y} f(\varepsilon_x,k_x,k_y)g(\varepsilon_x,k_x,k_y)d\varepsilon_xdk_xdk_y \quad [2]$$

Here $g(\varepsilon_x)d\varepsilon_x$ is the density of states with kinetic energy $\varepsilon_x$ within a unit volume, $f(\varepsilon_x)$ is the probability of an electron inhabiting those energy states, and $k_x$ and $k_y$ represents the electron wave-numbers in the y and z direction. To find $g(\varepsilon_x)d\varepsilon_x$, the phase space representation of the metal is considered, where the wave-number vector may be separated into its constituents:

$$\vec{k} = k_x\hat{x} + k_y\hat{y} + k_z\hat{z} \quad [3]$$

Where $k$ is the wave-vector of an electron.

In each unit volume of phase space, $(2\pi)^3$, there are two allowed states, one with electron spin up and one with electron spin down. Within the wave-vector range of $dk_xdk_ydk_z$, the number of allowed states with kinetic energy $\varepsilon_x$ is given by equation [4].

$$g(k_x,k_y,k_z)dk_xdk_ydk_z = \frac{2}{(2\pi)^3} \frac{\hbar^2}{m_e} dk_xdk_ydk_z \quad [4]$$

Here $m_e$ is the electron mass and $\hbar$ is Planck’s constant divided by $2\pi$. 

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Putting \( g(k) \) in terms of \( \varepsilon \) with the substitution suggested by equation [5] gives us equation [6]:

\[
\varepsilon = \frac{\hbar^2 k_x^2}{2m_e} \quad [5]
\]

\[
g(\varepsilon, k_y, k_z) d\varepsilon, dk_y, dk_z = \frac{2}{(2\pi)^3 \hbar} d\varepsilon, dk_y, dk_z \quad [6]
\]

The probability of an energy state being filled is given by the Fermi-Dirac probability distribution, equation [7]:

\[
f(\varepsilon) = \frac{1}{e^{\frac{\varepsilon - \varepsilon_F}{k_B T}} + 1} \quad [7]
\]

\[
\varepsilon = \varepsilon_F + \frac{\hbar^2 k_y^2}{2m_e} + \frac{\hbar^2 k_z^2}{2m_e} \quad [8]
\]

Here \( k_B \) is the Boltzmann constant, \( T \) is the absolute temperature, and \( \varepsilon_F \) is the metal's Fermi energy.

Inserting equation [6] and equation [7] into equation [2], making the substitution in the Fermi-Dirac distribution suggested by equation [8], and integrating over \( k_y \) and \( k_z \) gives:

\[
N(\varepsilon_x) d\varepsilon_x = \frac{4\pi m_e kT}{\hbar^3} \log(1 + e^{\frac{\varepsilon_x - \varepsilon_F}{k_B T}}) d\varepsilon_x \quad [9]
\]

Assuming the absolute temperature of the metal to be low enough that \( f(\varepsilon_x) \) looks like a step function with value 1 at \( \varepsilon_x \leq \varepsilon_F \) and a value of zero at \( \varepsilon_x > \varepsilon_F \), the following approximation can be made:
This results in a simpler form for \( N(\epsilon_x) d\epsilon_x \):

\[
N(\epsilon_x) d\epsilon_x = \frac{4\pi n e}{\hbar^3} (\epsilon_F - \epsilon_x) d\epsilon_x \quad \epsilon_x \leq \epsilon_F
\]  

[11]

To determine \( D(\epsilon_x) \), the tunneling probability of electrons with kinetic energy \( \epsilon_x \), consider the energy barrier at the surface, shown in Figure 1.

To escape, an electron inside the metal must contend with the difference in energy between its energy state in the metal and the energy level of the vacuum outside the surface. For the highest energy electron, at zero absolute temperature, this energy difference is the difference between the vacuum level and the Fermi energy. When a field \( F \) is applied at the metal surface, the vacuum energy barrier is reduced to a triangular shape described by equation [12] where \( x \) is the distance from the surface, as shown in Figure 1.

A more accurate model of the surface energy barrier takes into account the electron image force, which further alters the energy barrier as described in equation [13] and which is also shown in Figure 1.

\[
V(x) = E_{vac} - eFx
\]  

[12]

\[
V(x) = E_{vac} - eFx - \frac{1}{4\pi\epsilon_0} \frac{e^2}{4x}
\]  

[13]

Here, \( E_{vac} \) is the vacuum energy level and \( x \) is the distance from the metal surface.
The equation for the energy barrier is inserted into the one dimensional Schrödinger's equation to
determine the transmission probability coefficient, which yields the probability of an electron tunneling
through that barrier. An equation for the transmission probability coefficient for an arbitrary potential
barrier can be found by applying the Wentzel-Kramers-Brillouin\(^1\) approximation to the one dimensional
Schrödinger equation giving equation [14], where \(x_1\) and \(x_2\) are the zeros of the radicand with \(x_1 < x_2\).

\[
D(\varepsilon) = \exp\left(-\int_{x_1}^{x_2} \frac{8m_e}{\hbar^2} [V(x) - \varepsilon] \, dx \right) \tag{[14]}
\]

Inserting equation [12], representing the energy barrier observed by the electron in trying to escape the
metal surface, into equation [14], then inserting \(D(\varepsilon)\) and the \(N(\varepsilon)\) of equation [11] into the
original equation [1] and solving gives the Fowler-Nordheim result – equation [15]. If instead equation
[13] were used to represent the energy barrier, the more accurate, yet far more complicated set of
equations [16-22] results:

\[
j = \frac{e^3}{4(2\pi)^2} F^2 \exp\left(-\frac{4\sqrt{2m_e}\phi^{3/2}}{3\hbar eF}\right) \tag{[15]}
\]

\[
j = \frac{e^3 F^2}{8\pi\phi \hbar^2 (y)} \exp\left(-\frac{4\sqrt{2me\phi^{3/2}}}{3\hbar eF} v(y)\right) \tag{[16]}
\]

\[
v(y) = 2 \frac{1}{\sqrt{1 + \sqrt{1 - y^2}}} \left[ E(k) - (1 - \sqrt{1 - y^2})K(k) \right] \tag{[17]}
\]

\[
t(y) = v(y) - \frac{2}{3} y \frac{dv(y)}{dy} \tag{[18]}
\]

\[
K(k) = \int_{0}^{\pi/2} \frac{d\phi}{\sqrt{1 - k^2 \sin^2 \phi}} \tag{[19]}
\]

\[
E(k) = \int_{0}^{\pi/2} \sqrt{1 - k^2 \sin^2 \phi} \, d\phi \tag{[20]}
\]

\[ k^2 = \frac{2\sqrt{1-y^2}}{1+\sqrt{1-y^2}} \quad [21] \]

\[ y = \frac{\sqrt{e}F}{|\phi|} \quad [22] \]

In practice, equation [15] is the most commonly used equation to fit emission data, as there is less than a 5% difference between using equation [15] and the set of equations [16-22].

### 2.2 Local Field Enhancement Factor

The Fowler-Nordheim equation predicts that a field of \(10^7\) V/cm would be necessary to generate an emission current density of \(10^8\) A/cm\(^2\) from a tungsten tip. However, experimental emission data tends to be on the order of ten to one hundred times greater than the predicted emission current density. Schottky postulated that such an enhancement factor would be due to nanostructures on the tip surface. The geometry of these nanostructures concentrate the applied field locally and so they are locations of high electron concentrations. An example of this effect is shown in Figure 2. If a voltage is applied across two parallel plates separated by vacuum, the field lines will concentrate at small structures, commonly nanometer scale structures.

**Figure 2: Local field enhancement due to nanostructure.**

**Figure 3: Model for local field enhancement.**
To determine the effect of these nanostructures on the applied field, a model is necessary. One such model is a conductive sphere of charge $Q$ suspended above a flat metal surface. The sphere and the metal surface are connected by a thin conductive path – as in Figure 3. The sphere has radius $r$ and the conductive path has length $h$. An applied field between two opposing parallel plates creates a potential at a distance $h$ from the flat surface of:

$$\varphi = F_a h$$  \[23\]

If the charge $Q$ is assumed to be concentrated at the center of the sphere of radius $r$, the surface of the sphere has a potential due to the charge given by equation [24] and a field given by equation [25]:

$$\varphi = \frac{1}{4\pi\epsilon_0} \frac{Q}{r}$$  \[24\]

$$F = \frac{1}{4\pi\epsilon_0} \frac{Q}{r^2} = \varphi$$  \[25\]

Equating the two potentials represents connecting the sphere to the plate, then the substitution can be made:

$$F = \frac{\varphi}{r} = \frac{h}{r} F_a = \beta F_a$$  \[26\]

The apparent enhancement of the applied field is represented by the $\beta$ coefficient, which has been dubbed the ‘local field enhancement factor’. The local field enhancement can be considered a product of the nanoscale protrusion from the metal surface. The local field enhancement can be similarly produced by other nanostructures.

One example is found for flat metal surfaces coated with a thin insulator layer. If the insulator layer contains a matrix of thin conductive paths from the metal-insulator interface to the insulator-vacuum interface, local field enhancement can occur at each of these pathways. Our model for local field enhancement model is applicable to this situation as well. Here $h$ becomes the length of the conductive path.

---

paths and \( r \) becomes the radius of the conductive path at the insulator-vacuum interface. More example geometries and nanostructures found in the literature are listed in Appendix A.

To incorporate the local field enhancement factor into the previous equations, the substitution suggested by equation [26] is made. The Fowler-Nordheim equation becomes:

\[
j = \frac{e^3}{4(2\pi)^2} \frac{\beta^2 F_a^2}{h \phi} \exp\left(-\frac{4\sqrt{2m_e \phi^{3/2}}}{3\hbar e \beta F_a^2}\right) \tag{27}
\]

If the applied field is generated by placing a voltage across the emitting surface and a parallel plate that is held a distance \( d \) from the emitter, the Fowler-Nordheim equation can be rewritten as a relation between the applied voltage and the emission current:

\[
I = A_e \frac{e^3}{4(2\pi)^2} \frac{\beta V_a}{d} \left(\frac{\beta V_a}{d}\right)^2 \exp\left(-\frac{4\sqrt{2m_e d \phi^{3/2}}}{3\hbar e \beta V_a}\right) \tag{28}
\]

Here \( A_e \) is the area of the emitting metal surface.

In a field emission measurement system a metallic probe tip is used to apply a voltage to the sample. The metallic probe tip has a spherical geometry with a radius a few orders larger than the dimensions of emitting surface nanostructures. The probe tip is brought close enough to the surface that the probe tip can be approximated as plate parallel to the sample surface, allowing the use of equation [28] in fitting field emission data.

Along with the I-F curve, a ‘Fowler-Nordheim plot’ is generally shown for a material. Its linearity clearly illustrates whether or not the non-linear I-F curve can be represented by field emission. This is a plot of \( \ln(I/F^2) \) versus \( 1/F \). If the field emission data is properly described by the Fowler-Nordheim equation, the plot shows a straight line with a projected y-axis intercept of:

\[
b = \ln\left(\frac{A_e e^3}{4(2\pi)^2 \hbar \phi} \beta^2\right) \tag{29}
\]

And a slope of:
\[ m = -\frac{4\sqrt{2m_e\phi^{3/2}}}{3\hbar e \beta} \]  

A plot of the equation’s I-V curve with a workfunction of 5eV, a probe-sample distance of 5um, an emission area of \(10^{14}\) cm\(^2\), and a beta of 1000 is shown in Figure 4. It’s accompanying Fowler-Nordheim plot is shown in Figures 5.

![Figure 4: I-V curve of field-induced electron emission.](image1.png)  
![Figure 5: Fowler-Nordheim plot.](image2.png)

If the sample of interest has a relatively smooth surface, with no structures to contribute to a local enhancement factor, topographical data of the sample can be taken by field emission measurement equipment. As the probe is scanned over the sample, variations in the surface height will result in predictable variations in field emission current density. This information is translatable into topological data by use of the Fowler-Nordheim equation. However, because of the field emission current’s dependence on work-function, topological data is best taken of highly homogenous materials or materials with known work-function variations.
Section 3 System

A system was built to characterize the emission characteristics of nanostructured metallic surfaces. The system is currently capable of scanning a probe over the surface of a sample at a user specified distance, applying a field, and collecting emission current data. The data can then be exported for analysis, generally using a curve-fitting program to extract Fowler-Nordheim coefficients.

The system layout is presented in Figure 6 and a picture is presented in Figure 7.

![Diagram of system layout](image)

**Figure 6: System Layout**

The system consists of 3 subsystems:

- Vacuum Control and Maintenance
- Positioning (including Vibration dampening)
- Voltage Sourcing & Current Measuring

The experiment is conducted within a vacuum chamber maintained at a pressure of $10^{-6}$ Torr or better. The sample to be characterized is mounted into a holder within the vacuum chamber and a probe is brought close to the sample's surface. A nanomanipulation system is used to control the distance between probe and sample and to scan the probe over the sample surface. A voltage is applied between the probe tip and the sample and the field emission electrons generated by the sample are collected on the probe.
Either the voltage or the sample-probe distance is varied to obtain data for the dependence of emission current on field.

Figure 7: Picture of system
3.1 Positioning

**Figure 8: Diagram of probe stage and sample stage.**

**3.1.1 Probe & Sample Mounts**

The sample is mounted to an aluminum plate and held in place with sprung steel clamps and electrical contact is made with silver paint. The aluminum plate is attached to the metal sample base with nylon screws and nylon spacers, insulating the aluminum plate from the metal base. Electrical contact is made to the back of the sample through the aluminum plate or directly to the conductive sample surface with silver paint and copper wire. Figures 8 and 9 show a diagram and picture of the sample stage.

The probe is mounted to an XYZ positioning stage assembly of three stainless steel, spring loaded, linear translation stages that were modified for operation in high vacuum (NewFocus 9067-XYZ-R) by removing the fluorine based grease with PF5060 (3M) and replacing it with UHV lubricant. This assembly is capable of motion along the three perpendicular axes, relative to the assembly base. A three-dimensional diagram of the stage assembly is shown in Figure 10.

The complete sample holder and the complete probe system are fixed to an aluminum plate with screws. The aluminum plate is fixed to the bottom of the vacuum chamber. See Figure 15.
3.1.2 Probes

Two types of probes have been used with this system – a Molybdenum wire chemically etched to a sharp point or an AFM tip with an electrically connected gold coated sphere mounted to the apex of the AFM tip cantilever. These probes have tip diameters on the order of microns. The structures to be studied are generally on the nanometer scale. When the probe is brought close to the surface of the sample, the nanostructures see the probe tip as a virtual parallel plate capacitor. This allows us to use the approximation of equation [28] to fit field emission data.

The molybdenum wire used has a diameter of 0.5mm, a purity of 99.95%, and was obtained from Alfa Aesar. The etchant used is a mixture of 1 part ionized water, 1 part nitric acid (purity 69.9%), and 1 part sulfuric acid (purity 95%). Only the tip of the molybdenum wire is dipped into the etchant and it is kept there until the tip radius is 50um or smaller. This generally takes about half an hour. The molybdenum wire is then cleaned with compressed air and isopropanol. A magnified picture of the resultant probe tip is shown in Figure 11.
The AFM probe tips were purchased from BioForceNano. The tips come with a 5um diameter borosilicon sphere adhesively attached to the apex of each tip. The entire tip, including the sphere, is ion-beam sputter coated with 5nm of chromium followed by 10nm of gold with a resultant gold grain size of 5-10nm. A magnified image of the tip is shown in Figure 12. Conductivity from the base of the AFM tip to the sphere surface was not assured by the manufacturer.

![Figure 11: Etched molybdenum wire with 5um tip](image1)
![Figure 12: AFM tip with attached borosilicon sphere.](image2)

The AFM tip is mounted to the tip of a hard drive slider suspension spring using silver paint. The hard drive suspension spring is then attached by screw to an aluminum plate which is attached to the stage-assembly through a 0.5inch thick Teflon spacer for electrical isolation. See Figure 13 and 14.

A picture of the sample stage and probe stage mounted in the vacuum chamber is shown in Figure 15.

![Figure 13: Probe Mount](image3)
Figure 14: Probe mount and nanomanipulation system.
Figure 15: Sample and Probe nanomanipulation system mounted in vacuum chamber.
3.1.3 XYZ Stage and Motors

The position of each stage relative to the assembly base is controlled by a piezoelectric stepper motor (NewFocus 8302-V), see Figures 15 & 16. Within the stages are springs that maintain pressure against the piezoelectric motors’ screw tip so that when the screws move, the stages respond. A diagram of the piezoelectric motor’s operation is shown in Figure 17. When a voltage pulse is applied across the piezoelectric material, the threaded jaw slips around the screw and the screw does not turn. As the piezoelectric relaxes and the threaded jaws return to their original position, the screw turns, progressing the motor through one step and changing the position of the stage.

The motors produce a step size of less than 30nm that varies with the motor load. The load on the motors varies as the stages are moved and the stage springs are stressed. A plot of the step size versus motor load is shown in Figure 18. The motors have a velocity range of 1 to 2,000 steps per second and a total throw of 1 inch. The motors are connected to a Newfocus controller (NewFocus 8763 iPico Drivers) which is also networked to a computer outside the vacuum chamber through vacuum chamber feedthrough wires. Programming the motor motion is conducted either with a hand held touch pad (NewFocus 8757) or via the computer.

\[\text{New Focus.} \ (2001). \ Picomotor \ Drivers \ and \ Motorized \ Products \ Rev \ H. \ pp. \ 9\]
3.1.4 Laser Interferometer

A laser interferometer is used to measure the relative distance between the probe stage and sample stage. The laser interferometer consists of a dual wavelength helium-neon laser head (HP 5517C) operating at a beat frequency of 2.4MHz to 3.0MHz, a plane mirror interferometer (HP 10706A) with an attached quarter wave plate, a mirror attached to the back of the probe stage, and a receiver (HP 10780), see Figures 19 and 20. The interferometer uses interference patterns of two oppositely polarized laser beams to determine the relative position of the mirror from the interferometer cube. Calculation of the position is done within an HP Servo-Axis computer board (HP 10889B) by counting fringes and monitoring phase change as the mirror is moved. An HP supplied computer program is used to monitor the position readings of the interferometer. This interferometer system is capable of taking position measurements with a resolution of 2.5nm.

Figure 18: Piezoelectric motor step size as a function of load on motor.1

Figure 19: Diagram of laser interferometer system.

Figure 20: Laser interferometer system.
3.2 Vacuum System

The experiment is done in a vacuum chamber with a volume of 3600 in$^3$ (.059m$^3$). For this experiment a vacuum of $10^{-6}$ Torr or better is maintained. The vacuum chamber is first pumped down to a pressure of 1mTorr using a roughing pump. The roughing pump is then valved off and a cryopump (CTI Cryogenics Cryo-Torr 8F) is used to bring the chamber down to a pressure of $10^{-7}$ Torr, a process that takes less than a minute. Although the cryopump is able to stabilize a vacuum at better than $10^{-8}$ Torr (reaching a vacuum of $10^{-9}$ Torr after 36 hours) it introduces mechanical vibrations into the optical table and so into the experiment. Along the axis of the measurement, the cryopump generates 20um vibrations in the reading of distance between the probe and sample. For this reason, the cryopump is shut off during field emission measurements.
With the cryopump off, the vacuum chamber remains at a vacuum of better than $10^{-5}$ for approximately half an hour after which real and virtual leaks make the vacuum unusable for field emission. To maintain and stabilize the vacuum level, a differential ion pump (Gamma Vacuum DI Titanium and Tantalum 100L/S) is used. Once turned on, the ion pump maintains a vacuum of $5 \times 10^{-6}$. After 24 hours the pump can reach $10^{-6}$ Torr and after 36 hours levels off at $5 \times 10^{-7}$ Torr.

The ion pump itself also poses a difficulty to the field emission measurements. The ion pump generates free charge carriers that are ejected into the measurement chamber. The field used in the field emission experiment attracts these charged particles, they are collected on the probe mount and sample mount and appear as background current in the measurements. A plot of the current due to the ion pump collected at the field emission probe-sample setup versus the applied field between probe and sample is plotted in Figure 22. To reduce the background current a plasma shield was installed at the mouth of the ion pump. The plasma shield is simply a mesh of stainless steel wire with 2mm square openings and a wire width of 1mm. The mesh is grounded to the vacuum chamber and provides about a 20x factor background current reduction. A plot of the background current with the plasma shield installed is shown in Figure 23.

![Figure 22: Background current due to ion pump.](image1)

![Figure 23: Background current due to ion pump with plasma shield installed.](image2)
3.3 Voltage and Current Sourcing and Measuring

Voltage sourcing and current measurement are performed using the Keithley Sourcemeter 2400. The sourcemeter applies a voltage between probe and sample, regulating the voltage with an internal voltmeter. The voltage is swept through a user specified range and at each applied voltage the sourcemeter takes current measurements and stores them in its buffer, later to be collected by the computer. The electrical connections are sketched in Figure 24.

![Figure 24: Circuit layout.](image)

The total impedance of the connecting wires is approximately 0.4 Ohm. Initially, at low field-induced electron emission currents, the impedance of the system is determined by the impedance of the tunneling barrier, described by the Fowler-Nordheim equation. Once this value falls below the total resistance of the connecting wires and the resistance of the probe, the value of I/V saturates to this combined resistance.

A Visual Basic program with a graphical user interface (GUI) is used to sweep the voltage output of the sourcemeter which is applied between the probe and sample in a linear ramp. Values of current at each applied voltage are collected in the sourcemeter and, once the voltage sweep is complete, the data is sent to the computer to be plotted. The GUI currently allows for five different types of measurements:
• The Full Field Emission function - sweeps through a voltage range in uniform increments, both the range and the increment values being specified by the user, taking the average of 20 current measurements at each voltage with a 16.7 ms integration time per measurement.

• The Field Emission Hysteresis function - performs The Full Field Emission function with repeated positive and negative voltage sweeps through the voltage range.

• The Constant Voltage function - applies a constant voltage and measures current values over time, also using the same A/D integration time (16.7ms) and the same averaging filter (20 samples).

• The Fast Field Emission function - sweeps through a user specified voltage range using 10 equal voltage increments using a 1.67ms A/D integration time and single measurement values, generating a field emission plot in a much shorter time than the Full Field Emission function, but at a reduced resolution.

• The One Point Emission function applies a short term voltage to the sample and measures the current present between the rise and fall time of the applied voltage. This function is used to ascertain if the probe is making contact to the sample surface. The voltage is applied for 2ms and the A/D integration time used is only 167us.

The A/D integration time impacts the resolution of the current readings. At current measurement ranges of 10uA and 100uA, the various current resolutions for varying A/D integration times are shown in Table 1. An integration time of 16.7ms corresponds to an integration of the power line noise over one power line cycle (60Hz).

<table>
<thead>
<tr>
<th>A/D Integration Time</th>
<th>Current Resolution at 10uA Meas. Range</th>
<th>Current Resolution at 100uA Meas. Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>16.7ms</td>
<td>0.5nA</td>
<td>5nA</td>
</tr>
<tr>
<td>1.67ms</td>
<td>5nA</td>
<td>50nA</td>
</tr>
<tr>
<td>0.167ms</td>
<td>50nA</td>
<td>500nA</td>
</tr>
</tbody>
</table>

Table 1: A/D Integration time verse current measurement resolution.

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Section 4  Method

The probe to be used is mounted to the nanomanipulation stage. The sample is mounted onto the sample holder, the sample holder is aligned to the probe stage so that the x-axis of the probe stage is perpendicular to the sample surface, and the sample holder is fixed to the aluminum base plate. The vacuum chamber is held at atmospheric pressure and the valved off cryopump is electrically shut off to reduce mechanical vibrations. The probe is brought into visible contact to the sample by using the hand held touch pad and an I-V curve is taken using the Visual Basic GUI. This I-V curve is used to compare with I-V curves taken under vacuum pressure to determine when the probe is in contact with the sample.

The vacuum chamber must now be pumped down to $10^{-6}$ Torr, however the mechanical vibrations induced into the experiment by the roughing pump and the cryopump make it necessary that the probe is backed off a sufficient distance so that the probe tip does not hammer into the sample surface. Once the probe is backed off from the sample surface, the valved off cryopump is turned back on. The vacuum chamber is pumped down to $10^{-3}$ Torr with the roughing pump, the roughing pump is valved off and the valve isolating the cryopump is opened to bring the vacuum chamber to $10^{-8}$ Torr. The ion pump is now turned on and given time to stabilize it's operational voltage between 6000 and 7000V. The cryopump is now shut off (with it's valve left open) and the ion pump is allowed to stabilize the vacuum chamber pressure to $10^{-6}$ Torr.

The probe is brought toward the sample by eye, through a vacuum chamber viewing port. The sourcemeter is turned on and the probe is slowly brought toward the sample surface. The sourcemeter is set to a constant voltage and the emission current is watched for a value that hints at contact. When probe-sample contact is suspect, an I-V curve is taken and compared to the initial contact I-V curve for verification. If contact has been made, the position of the surface has been determined and the laser interferometer position reading is reset to zero. The probe is backed off a user specified distance using
the laser interferometer reading. At this point the Full Emission Curve program can be initiated to collect field emission data. When the field emission measurements are complete, the voltage and current data are recorded and plotted on the PC screen.
Section 5  Performance Analysis

5.1 Field Emission Measurements

The two key components of the field emission measurement are the sample-probe distance measured via
the laser interferometer and the applied voltage controlled and measured by the sourcemeter. Each of
these has a resolution rating and a noise rating, which contribute to an expected error in accuracy and a
noise level in the current measurements. Here resolution is taken to mean the smallest portion of the
voltage that can be sourced or the smallest portion of the current that can be measured and displayed.
Resolution is determined by the instrumentation. The noise level is defined as any unwanted signal that is
imposed on the signal being sourced or measured. In this case, noise in the interferometer reading is due
to temperature and pressure variations as well as mechanical vibrations along the measurement path.
Noise in the voltage sourced is expected to be due to line noise and thermal variations in the circuit. An
estimate of the expected error in accuracy and the expected noise level in the current measurements
follows.

The individual contributions of position and voltage to the error in field is determined from $F = V/d$ as:

$$\Delta F = \frac{1}{d} \Delta V - \frac{V}{d^2} \Delta d$$  \[31\]

This relation is used to determine the expected accuracy and noise of the current measurements in the
Fowler-Nordheim equation:

$$\Delta I = \frac{\partial I}{\partial F} \Delta F = A_c K_\phi \beta^2 \left[ 2 F + \frac{K_3 \phi^{1.5}}{\beta} \right] \exp\left( - \frac{K_3 \phi^{1.5}}{\beta F} \right) \Delta F$$ \[32\]

By inserting the instruments’ accuracy ratings into these equations the expected error in accuracy
for the current measurements is obtained; by inserting the instruments’ noise levels the expected
noise level in the current measurements is obtained.
For the most commonly used voltage range (0-200V) the expected noise is 5mV and the expected error in accuracy is 0.02%*V + a 24mV zero offset. The observed noise of the laser interferometer is 100nm and the expected resolution of the laser interferometer is 2.5nm. Inserting these values into equations [31] and [32], with an applied voltage of 50V and a sample-probe distance of 5um results in the relationship between beta and the log of current density accuracy as well as the relationship between beta and the log of current density noise plotted in Figure 25. Points of interest are shown in Table 2. For each beta, the max emission area, A_e, allowable before the error reaches 10^{-10} Amps is listed.

Figure 25: Current Density Error and Noise versus beta. Table 2: Expected accuracy and noise levels.

At an A/D integration time of 16.7ms, the sourcemeter displayed resolution is 0.5nA. In the range typically used for current measurements (10uA), the sourcemeter accuracy rating is 1\%0.027\%+700pA, at a current of 10uA that gives an accuracy error of 3.4nA. So the limiting measurement for samples with beta’s up to 1000 is set by the sourcemeter’s accuracy in current measurements.

If the level of noise in the interferometer were lowered by an order of magnitude to 10nm, the expected current density noise level would also decrease by an order of magnitude, giving the results shown in Table 3.
<table>
<thead>
<tr>
<th>$\beta$</th>
<th>$10^2$</th>
<th>$5 \times 10^2$</th>
<th>$10^3$</th>
<th>$10^4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Delta J_{\text{noise}} [\text{A/cm}^2]$</td>
<td>$10^{-26}$</td>
<td>6</td>
<td>$3 \times 10^4$</td>
<td>$10^9$</td>
</tr>
<tr>
<td>Max $A_c [\text{cm}^2]$</td>
<td>$10^{16}$</td>
<td>$2 \times 10^{-11}$</td>
<td>$3 \times 10^{-15}$</td>
<td>$10^{-19}$</td>
</tr>
</tbody>
</table>

Table 3: Expected accuracy and noise levels with interferometer noise level of 10nm.
5.2 Probes

5.2.1 Molybdenum Probe

For the sharpened molybdenum tip, the main concern is the mechanical softness of the tip. While collecting field emission measurements the probe must make contact to the sample to determine the zero reference probe-sample distance. With repeated probe-sample contact the molybdenum tip deforms. The sharp tip becomes flattened and eventually curls back on itself, See Figure 26. Because of this deformation, the molybdenum tip was determined to be a poor probe choice for repeated experiments even though it has been reportedly used with good results in the literature.

![Figure 26: Blunted Molybdenum probe.](image)

5.2.2 AFM Probe

The AFM tips as purchased were not assured to be conductive from the base of the AFM tip to the sphere surface. However, resistances on the order of 1Mohm were commonly observed. This resistance greatly limits the ability of the apparatus to measure the field emission curve of a sample. As the tunneling current increases with increasing applied voltage and the sample surface’s resistivity decreases and approaches that of the probe tip, the resistivity of the probe tip becomes the dominant resistance in the

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system circuit, masking the I-V characteristics of the sample. Higher conductivity probe tips can be purchased as custom made from BioForceNano and will be tested in the future.

With a probe diameter of 5um and a sample-probe distance typically on the order of 1um, bending of the AFM beam due to the electrostatic force is possibly a serious concern. This beam bending decreases the actual probe-sample distance with increasing field and can potentially result in the probe snapping to the sample surface. The expected probe tip deflection due to the electrostatic force was determined by measurements of the beam deflection due to a force applied at the tip. The relation between the force applied to the tip and the tip deflection was empirically found to be (see Figure 27):

\[ y = 31.5 \left( \frac{s^2}{kg} \right) \cdot F_e \]  

[33]

Where the constant is equivalent to the inverse of the spring constant in Hooke’s Law. The electrostatic force at voltage \( V \) and sample-probe distance \( d \) is calculated from the following equation:

\[ F_e(d, V) = \frac{1}{2} \frac{\partial C(d)}{\partial x} V^2 \]  

[34]

For the capacitance between a conductive sphere and a metal plane, the capacitance is given as:

\[ C(d) = 4\pi \varepsilon_0 r \left( 1 + \alpha + \frac{\alpha}{1 - \alpha^2} + \frac{\alpha^3}{(1 - \alpha^2)(1 - \alpha^3)} \right) \]  

[35]

Where \( \alpha = \frac{r}{2(r + d)} \)  

[36]

Where alpha is given in equation [36], \( r \) is the radius of the sphere and \( d \) is the shortest distance from the sphere’s surface to the metal surface. A plot of the expected electrostatic deflection as a function of probe-sample distance is shown in Figure 28. At the common sample-probe distance of 5um and an

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applied voltage of 50V, the expected deflection is 9.8A. At a sample-probe distance of 100nm (or larger) and an applied voltage of 50V (or less), the expected deflection is 89A (or less). Beam deflection due to electrostatic force is not a significant concern until the probe-sample spacing goes below 1nm.

![Graph](image)

**Figure 27:** Beam tip deflection versus force on tip.

![Graph](image)

**Figure 28:** Expected electrostatic deflection versus probe-sample distance.

Excessive high field emission current is another concern of using the AFM probe tip. At field emission currents of $10^{-4}$A the probe tips will show serious thermal damage and the borosilicon sphere will disappear. See Figures 29 and 30. This dictates that the emission current should be limited at the supply (the Sourcemeter) to less than or equal to $10^{-5}$A.
5.3 Laser Interferometer

The HP laser interferometer system has a position measurement accuracy of 2.5nm. However, due to environmental conditions, the interferometer currently operates with a peak to peak noise of approximately 100nm. These environmental concerns are primarily air pressure variations along the laser beam’s path and mechanical vibrations. This noise level is acceptable for sample-probe distances on the order of a micron, but with a motor resolution at better than 30nm, one would wish for an interferometer resolution of an order of magnitude less to take full advantage of the step size of the motors. In an attempt to reduce the noise due to air pressure variations, a box was placed over the entire laser interferometer system blocking air currents from interacting with the laser beam. However, this resulted in no significant reduction in the noise levels. It’s believed that vibrations from the environment are keeping the system from its optimal operation. The vacuum chamber and interferometer system are mounted to an optical table with vibration isolator legs, which are currently the primary means for vibration dampening. The vibration isolator legs (Melles Griot SuperDamp Self-Leveling Vibration Isolators) have a vibration transmissibility shown in Figure 31. At frequencies above 1Hz the legs reduce vibration transmission by one to two orders of magnitude.
Figure 31: Vertical and horizontal transmissibility of SuperDamp isolators.
Section 6  Example Results

6.1 Platinum

A 0.5mm thick piece of platinum was tested for field emission properties. No field emission was detected with a minimum probe-sample spacing of 100nm. This was expected as the Platinum sample has a very smooth surface. Assuming an enhancement factor of 1, an emission area of $10^{-10} \text{cm}^2 \ (10^4 \text{ nm}^2)$, and a workfunction of 5.5eV, to achieve a current of $10^{-10}$ Amps a field of 4000V/um is needed – a field that the current system is not capable of producing.

6.2 Carbon

A carbon film deposited on a conductive silicon wafer was tested. The carbon film was generated with the use of the self-assembly process of block copolymer chemistry, deposited using the zone casting technique\(^1\), and then pyrolyzed at 800degC. Figure 32 shows the I vs. V curve of the material as well as a Fowler-Nordheim curve fitting; Figure 33 shows the Fowler-Nordheim plot of this data.

Figures 32 & 33: I vs F and FN plot for carbon film. FN fitting with workfunction held at 5eV.

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The curve fitting was done with the probe-sample distance parameter set to the measured 2\,\mu m. The workfunction parameter was set to 5\,eV, a very common workfunction value for nanostructured carbon films. The curve fitting (the red curve in Figure 32) resulted in an enhancement factor of 650 and an emission area of $6.7 \times 10^{10} \, \text{cm}^2$ ($6.7 \times 10^4 \, \text{nm}^2$), both really good values. Literature values of the best enhancement factors from nanostructured carbon generally fall between $10^2$ and $10^3$; emission areas are typically between $10^2 \, \text{nm}^2$ and 1 \,\mu m$^2$.

Furthermore, the Fowler-Nordheim plot shows a “turn-on” field of 6.7 \,\mu V/\mu m, also a fairly good value, falling in the desired range of 1 \,\mu V/\mu m to 10\,\mu V/\mu m. The turn-on value is described as the field at which the I-V curve begins to exhibit a Fowler-Nordheim behavior. It is found from the Fowler-Nordheim plot as the field at which the plot shows a kink.

### 6.3 Hydrogenated Amorphous Carbon (Diamond Like Carbon)

A thin film of hydrogenated amorphous carbon was deposited on a Nickel substrate by sputtering. Field emission was detected at a probe-sample spacing of 1\,\mu m. Figures 34 and 35 show the I-V Fowler-Nordheim curve fitting and the Fowler Nordheim plot, respectively. A Fowler-Nordheim curve fitting with the assumed 5\,eV workfunction resulted in an enhancement factor of 8000 and an emission area of $8 \times 10^{-20} \, \text{cm}^2$. Neither the emission area nor the enhancement factor seem reasonable. This is most probably due to an incorrect assumption of workfunction. When the curve fitting is repeated with the workfunction parameter set to 1\,eV a more reasonable enhancement factor of 700 results. However, the resultant emission area still seems implausible at $2 \times 10^{18} \, \text{cm}^2$ ($2 \times 10^2 \, \text{\AA}^2$). See Figure 36. The workfunction of this thin film should be obtained through other means and used as an input parameter for the Fowler-Nordheim curve fitting. The Fowler-Nordheim plot shows a turn-on field of approximately 8\,\mu V/\mu m.
This set of data from the platinum, the nanostructured carbon and the diamond like carbon look promising and the nanostructured carbon appears to be a good emitter choice. To facilitate future work, a means should be obtained for gathering workfunction information on samples.
Section 7  Future Work

While the system performs pretty well, much can be done to improve it via probe choice, nanomanipulation control, and background current reduction. Molybdenum turned out to be a poor choice for a probe metal because of its softness. Other types of metals, preferably harder than molybdenum, should be tried along with different wire sharpening techniques. An attempt should be made at making the wire probe tips as spherical as possible for symmetry purposes.

The AFM tip currently used has the optimal gold coated, spherical contact surface but is too resistive. Either a more conductive AFM tip should be purchased or the current tips used could be sputter coated with additional gold to increase surface conductivity. Tip warping due to film stress will be a concern.

Currently the background current due to the ion pump is either subtracted out from emission data or the ion pump is turned off during data collection. A system could be constructed to reduce the ion pump current. One possible system would work to accelerate these particles to be collected at electrodes located between the ion pump and the field emission setup, reducing the amount of electrons and ions that make their way to the experiment.

The nanomanipulation system could be greatly improved by writing a computer program to control probe-sample distance as the voltage is increased. This would make taking field emission curves with varying sample-probe distances a far easier task. If the program uses the HP Servo-Axis card to control the piezoelectric motors, a feedback loop can be made with interferometer distance measurements and probe movement control. This may provide much more precision and a simpler interface in probe positioning. Further integration of the Keithley sourcemeter with probe positioning would result in one interface for the complete field emission characterization system.
Fig. 8. Various structures proposed for LMF emitters based on non-carbon films and PGL carbon films, classified using C/D/V terminology. Except for Fig. 8(i), diagrams are reproduced or adapted from the references shown.