

Characterizing the Electro-Optic Properties of a Microfabricated Mass Spectrometer

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1. Abstract

Today, mass spectrometers are expensive and cumbersome, and they cannot be used outside of the laboratory environment. However, advances in Microelectromechanical Systems (MEMS) technology, carbon nanotube growth, and computational spectroscopy make possible a new class of miniature sensing systems. We are currently developing a microfabricated mass spectrometer with the objective of deploying it to a hand-held device. Such a device could have applications in defense, medicine, and space by allowing portable chemical analysis. A micro mass spectrometer would also benefit from reduced power consumption and low cost, making it an even more compelling tool for use outside of the laboratory.

This work focuses on the development and use of experimental tools used for characterizing prototypes of the microfabricated mass spectrometer. We report the design and implementation of hardware and software tools that characterize different parts of the device. Integrating and automating these tools with National Instruments' LabVIEW programming environment has reduced manual work, enabled easy data collection, and increased experimentation speed.

The first part of the project was developing LabVIEW software to perform characteristic tests for prototypes of the micro ion source, the first component of the microfabricated mass spectrometer. We performed diode and triode characterization with LabVIEW to demonstrate basic functionality in a prototype of the ion source. In a second part, software for analyzing the ion beam emanating from an ion source is presented. We demonstrate that the ion beam imaging system that has been developed can detect gases and can, therefore, be used to analyze mass separated ion beams. Improvements on the imaging software, including noise removal and flattening, are also discussed.

2. A Microfabricated Mass Spectrometer

1. An Impact Ionization Mass Spectrometer

Impact ionization mass spectrometry contains two key parts: an ion source and a magnetic sector. The ion source produces electrons that impact ionize the gaseous analyte by knocking out electrons of the atoms or molecules of the gas. The resulting positively charged ions are accelerated out of the ion source by an electric field. The ion beam then enters the magnetic sector where a constant magnetic field gives each ion a circular trajectory whose radius depends on its mass to charge ratio. The deviated ions strike at different distances along a horizontal ion detector: ions whose mass to charge ratio is larger land further along the detector. Ions of the same mass to charge ratio strike the detector at a unique point where they produce electric current. Current measurements along the detector result in a plot of current versus distance that can be transformed into a current versus mass to charge ratio plot, a mass spectrum. Analysis of current intensity peaks in mass spectra determines the composition of a gaseous sample. The functional diagram of an impact ionization mass spectrometer can be found in Figure 1.

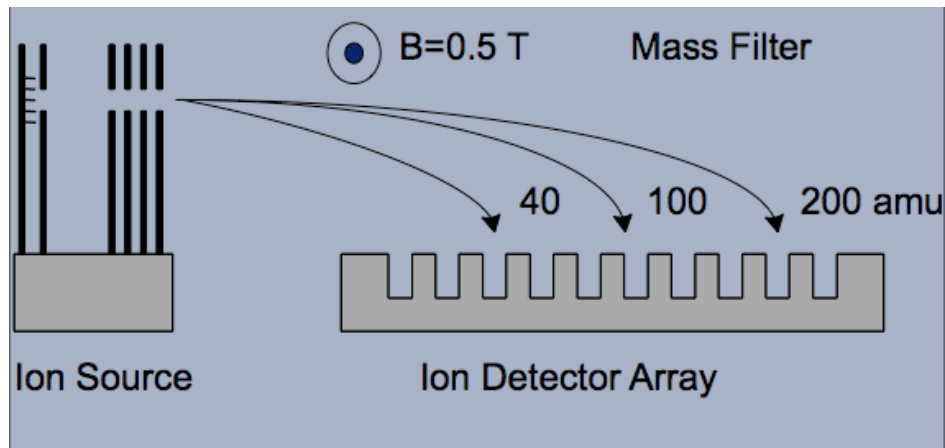


Figure 1: Diagram of an Impact Ionization Mass Spectrometer

2. A Microfabricated Ion Source with Carbon Nanotube Cold Cathode

A microfabricated ion source with carbon nanotube field-emitters impact ionizes the analyte while miniaturizing device dimensions and minimizing the need for power dissipation. The device's features are presented in Figure 2. The ion source is fabricated via MEMSCAP's POLYMUMPS¹. Carbon nanotubes that are used for producing electrons are grown in bundles on the cathode using a Plasma Enhanced Vapor Deposition (PECVD) system. Incorporating into the process flow the growth of a 10-micron thick layer of silicon dioxide at the base of the panels enables voltages as high as 800 V to be applied to an individual panel². Through wire bonding, the panels are connected to exterior voltage sources. The panels are then flipped up and locked into a vertical position so that the electric fields emanating from them create a potential field that moves charged particles.

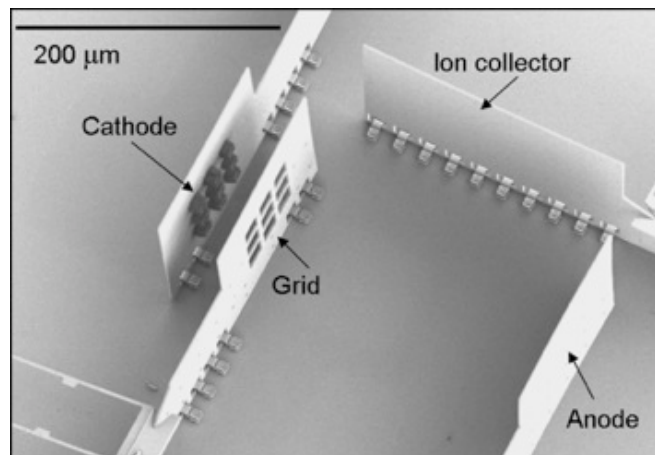


Figure 2: SEM micrograph of a micro ion source with a carbon nanotube cold cathode

The ion source uses carbon nanotube field emission as its electron emission process. This offers an alternative to traditional thermionic emission, where a metal filament is heated up to emit electrons. This method results in high device temperatures that impose a significant thermal budget. Carbon nanotube Fowler-Nordheim field emission is a quantum mechanical effect that produces no heat. When a voltage above a

certain threshold is applied across the cathode and grid panels in Figure 2, electrons tunnel through the carbon nanotube tips and are electrostatically attracted towards the electron extraction grid.

Holes in the grid allow the electrons to pass through it and enter the area between the grid and anode. In this area, the electrons impact ionize the gas, with the highest probability of ionization occurring at electron energies in the range of 70 to 100 eV³. Applying a high positive voltage to the anode attracts the electrons through this area, ensuring that ionization events occur throughout the ion source. Replacing the ion collector panel in Figure 2 with a series of Einzel lenses in the form of micro-machined panels accelerates the positively charged ions out of the device in the form of an ion beam.

3. Magnetic Sector

The ion beam enters the magnetic sector where the ions are first separated according to their mass to charge ratio, if we assume that they have been accelerated to the same speed by the Einzel lenses. The ions then land on a horizontal ion detector. The constant magnetic field in the magnetic sector makes the ions adopt a circular trajectory, as illustrated in Figure 3. Equations (1) describe the relationship between the strike distance of an ion and its mass to charge ratio.

$$d = \sqrt{2rh - h^2} \quad r = \frac{1}{B} \sqrt{\frac{2mE}{q}} \quad (1)$$

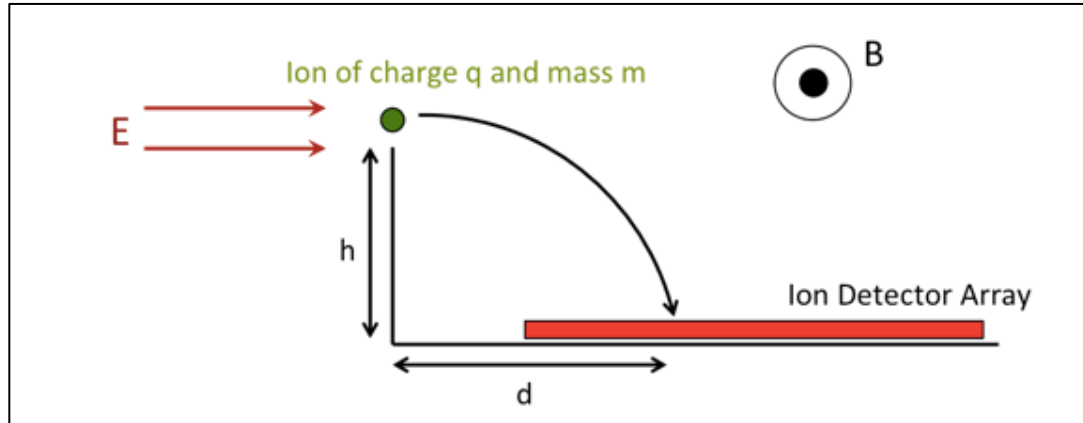


Figure 3: Diagram of the Magnetic Sector

For every ion, the radius of the circular path is a non-linear but monotonic function of its mass to charge ratio: the higher the ratio, the larger the radius is. The ion beam is thus spread (or “separated”) according to the mass to charge ratio of the ions, and this spread manifests itself when the ion beam lands on the ion detector. By taking current readings along the length of the detector, a plot of current versus strike distance is obtained. Using Equations (1), the mass to charge ratio is related to strike distances so that the aforementioned plot is converted into a mass spectrum, which gives information about the composition of the gas.

3. Experimental Setup for testing a Mass Spectrometer

1. Vacuum Chamber Setup

Prototypes of the micro ion source are tested in a vacuum chamber. A relatively low vacuum between 10^{-5} and 10^{-7} Torr is necessary to ensure that the flights paths of the electrons and the ions are not obstructed by collisions with stray particles. However, the minimum vacuum required is relatively low because of the small device dimensions, on the order of hundreds of microns. The dimensions are close enough to the mean free path of electrons at the relatively low vacuum of 10^{-5} Torr that impact ionization still occurs at those pressures. A picture of the vacuum chamber is shown in Figure 4. It features feedthroughs that allow electrical connections between power supplies outside of the vacuum chamber and the micro ion source. This means that the voltages applied to the panels on the device can be controlled while the chamber is pumped down.

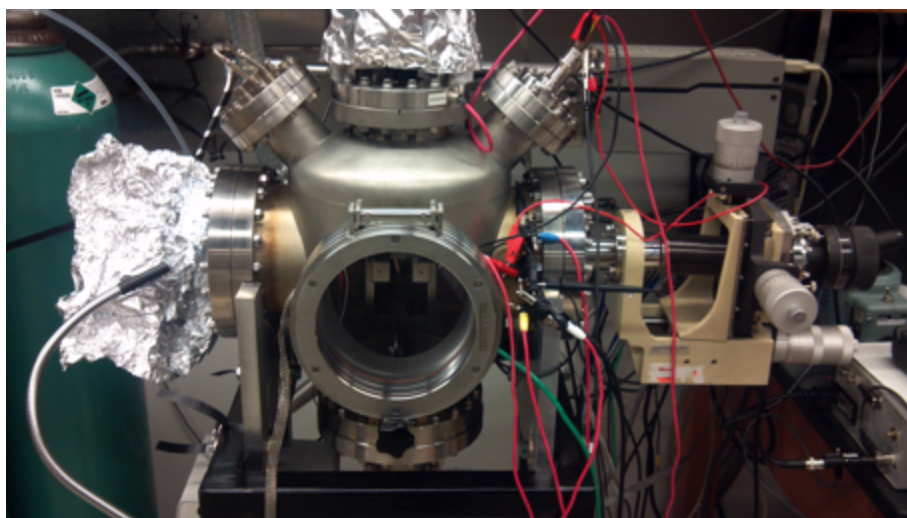


Figure 4: Vacuum Chamber

2. Power Supplies

We use a Keithley 2400, a Keithley 2410, and a Keithley 6487 sourcemeter to apply voltages anywhere from -1000 to +1000 V to the panels of the micro ion source. These power supplies are very useful because they can source a voltage and measure current across whatever they are powering. Additionally, these sourcemeters can also be controlled with a computer using the IEEE-488 communication protocol⁴, also known as General Purpose Interface Bus (GPIB).

Additionally, we have designed an array of Spellman MPS power supplies⁵. These low-cost voltage sources are useful for applying high voltages of up to 3 kV to components of the micro ion source or the experimental setup. Up to eight individual Spellman MPS units are mounted into a rack-mount that enables safe and easy use of these high voltage parts. The hardware array is shown in Figure 5. A main switch on the right of the front panel controls power to whole piece, while eight switches allow

the user to turn on or off individual MPS units. The array is also connected to a National Instruments PCI analog output card that outputs eight 0-10 V analog control signals that are each connected to the “Voltage Program Input” pin of one of the eight MPS units in the array. That way, the voltage of each individual MPS unit can be controlled from a computer.



Figure 5: Spellman MPS Array

3. Ion Beam Detector

The ion beam from the ion source can be visualized in real time with the Beam Observation System (BOS) from Beam Imaging Solutions⁶. The BOS consists of a micro-channel plate (MCP), a phosphor screen, and a high-resolution camera, the SONY XCD-U100. The BOS acts as an ion beam detector and is placed perpendicular to the deviated ion beam in the magnetic sector to obtain a cross section of the beam, as seen in Figure 6. On the MCP, electron multiplication microchannels amplify current resulting from ions striking their inner walls. The produced electrons are accelerated towards the phosphor screen, which is powered by a Spellman MPS. An electron landing on the phosphor is then converted into photons, which can be visualized by the high-resolution camera taking a digital image of the phosphor screen.

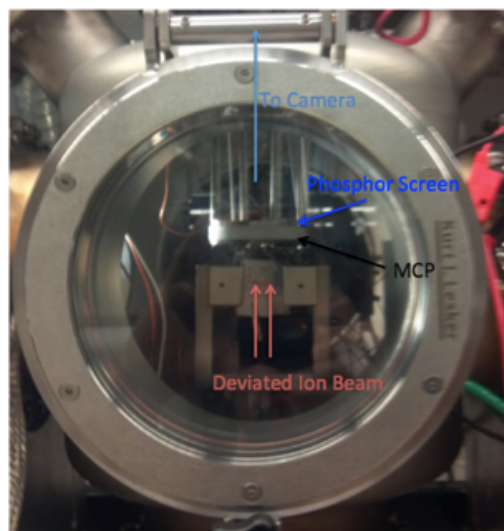
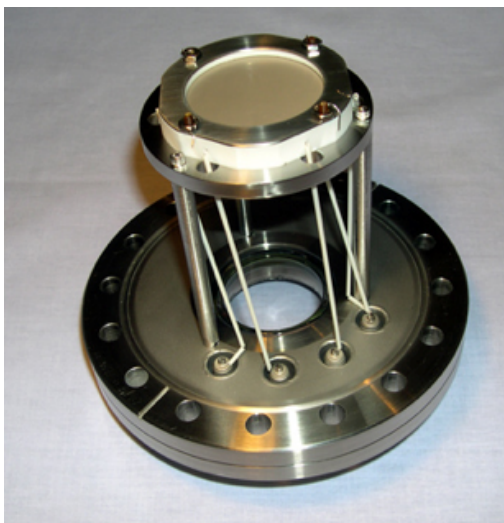


Figure 6: Ion Beam Detection System L: BOS R: BOS in vacuum chamber

4. Characterizing the Micro Ion Source

1. Diode Characterization

1. Diode Characterization

Diode Characterization quantifies how well electron currents can be extracted from the carbon nanotubes in the micro-ion source. The produced electrons will subsequently impact ionize gases in the device. The diode consists of the cathode and the electron extraction grid panels in the micro ion source. An SEM micrograph of the diode can be seen in Figure 7.

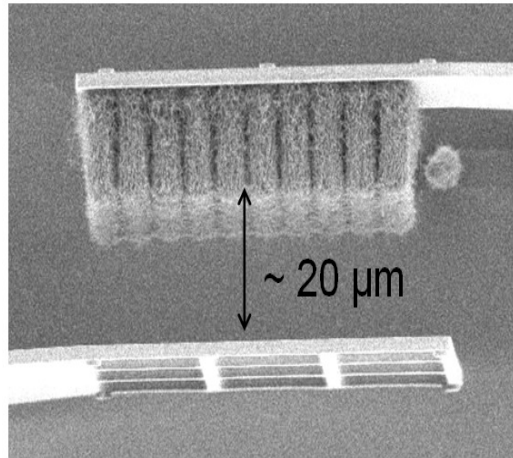


Figure 7: SEM micrograph of the Diode (top panel: cathode, bottom: extraction grid)

Diode Characterization consists of holding the cathode voltage constant while sweeping the electron extraction grid voltage. As the voltage between the two panels increases, the electric field in the area between the two panels increases. Beyond a certain voltage threshold, we expect the carbon nanotubes of the cathode to field emit electrons through the Fowler-Nordheim field emission process. The electrons will subsequently be electrostatically accelerated towards the electron extraction grid, where some would create current upon impact, while others would pass through its holes. As a result, current on the grid is expected to increase in magnitude as the voltage on the grid is increased.

2. Experiment and Results

The cathode was held at a voltage of -100 V while the electron extraction grid was swept from 0 to 200 V seven times. We set the cathode to a negative voltage so that it would electrostatically repel any electrons the carbon nanotubes field emitted. Plots of the grid currents versus grid voltages for the seven data runs are presented in Figure 8.

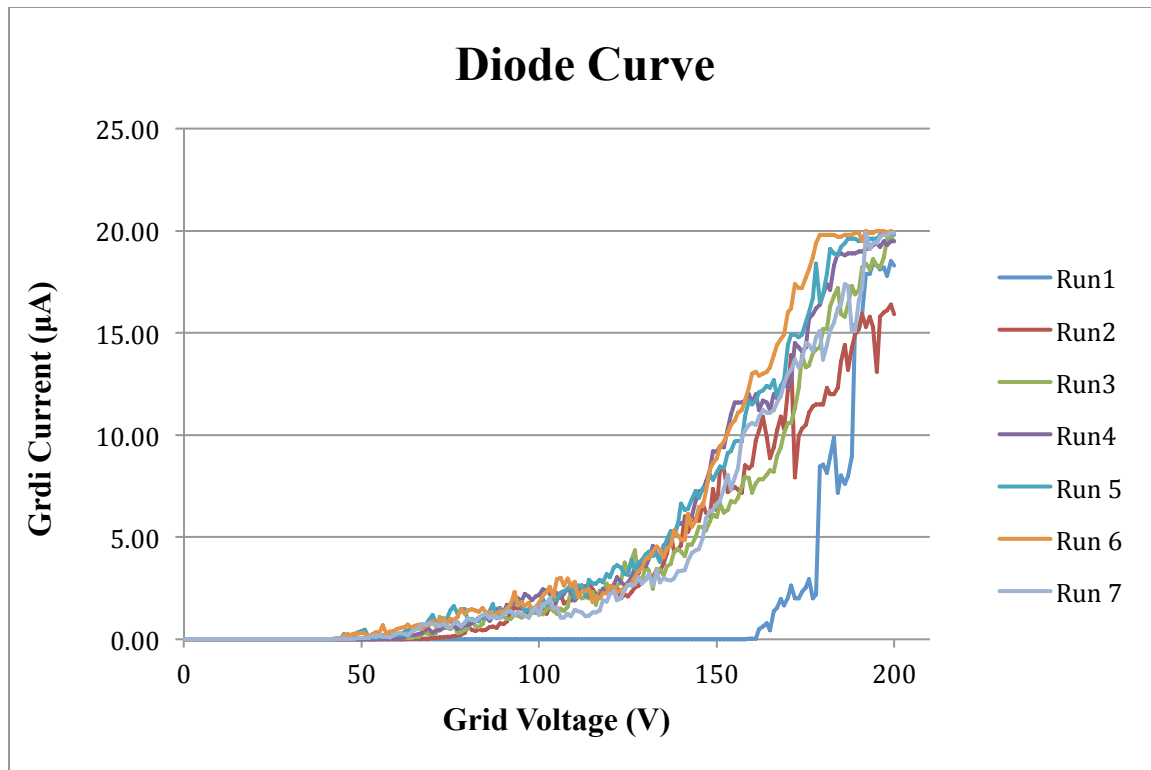


Figure 8: Diode Characterization Curve

The current on the grid increases with the bias between the grid and the cathode. For Run 1, the current begins to increase at much higher voltage than the other runs. We suspect that this is due to the presence of adsorbates on the carbon nanotubes when the diode is first turned on. Once the two first sweeps are complete, the adsorbates have been removed and the diode exhibits very similar behavior for all subsequent runs.

The Keithley Sourcemeters that were used to run this experiment were set at a current compliance of 20 μA , which justifies why the grid current does not increase past 20 μA . In reality, we expect the current to continue increasing past this value as the grid voltage increases. The exponential relationship between grid current and grid voltage in the non-compliance range confirms that Fowler-Nordheim field emission is occurring, with a turn on voltage of 160 V for all but Run 1. This shows that the diode is producing electrons that are emitted from the carbon nanotubes before landing on the electron extraction grid.

3. LabVIEW VI diode_characterization.vi

This LabVIEW Virtual Instrument (VI) was coded to perform the diode characterization tests. The user first inputs the voltage at which the cathode is held and the range of voltages the electron grid sweeps over. The power supplies to use are chosen from a drop-down box for the cathode and the extraction grid. The user then chooses to perform a linear or logarithmic sweep. The experiment can automatically be performed as times as wanted. A name for the data run can be entered, as well as any notes that describe the experiment.

When the user launches the VI, the experiment begins. The VI communicates with the power supplies for the cathode and the electron extraction grid by sending Standard Commands for Programmable Instruments (SCPI) commands through the GPIB interface. It first prepares them to source voltages and measure current, sets the voltage for the cathode, and then sweeps the voltage for the electron extraction grid while measuring the current on both power supplies between each point in the sweep. A problem encountered in the earlier version of this code was that the power supplies did not measure current at the same time. In earlier versions of the code, two “READ” SCPI commands were sequentially sent to the Keithley sourcemeters to sequentially trigger a measurement and return the value to the computer. This proved to be too slow: because one Keithley returned its measurement value to the computer before the other took its measurements, one could visually observe the lag between the measurements on the digital displays of the instruments. To reduce this lag in the final version of the code, the two sourcemeters are set up before the experiment to take a measurement and store it into their buffers when receiving an “INIT” command. That way, two “INIT” commands are sequentially sent to the sourcemeters when a sweep point has been reached to trigger the measurement. Once both devices have performed their measurement, the value of each measurement is returned to the computer with two sequential “FETC?” commands, which fetch the values of the measurements out of the buffers in the devices. The use of these low-level commands helped synchronize the sourcemeters.

When the sweeps have been completed, all the data is saved into an .txt file that bears the name the user gave to the experiment. The notes the user took are also placed at the top of this file. This file is automatically placed into a folder that has the date of the experiment as its name. This way, data is automatically organized in such a way that it is easy to find and to begin processing it.

2. Triode Characterization

1. Triode Characterization

Triode Characterization quantifies how well the anode can attract the field emitted electrons through the ion source. Accelerating the electrons through the ion source is necessary to impact ionize the neutral particles in the area between the grid and the anode. Figure 9 presents an SEM micrograph of the ion source. Figure 10 summarizes the function of the micro ion source, with an additional ion collector panel.

In triode characterization, current on the anode is measured as the cathode is held at a constant voltage, the electron extraction grid steps voltages, and the anode sweeps voltages. For low grid voltages, the diode should not be field emitting, so we expect the anode current to remain low throughout the anode sweep. For grid voltages past a certain threshold, the diode will be field emitting, so we expect anode current to increase with anode voltage.

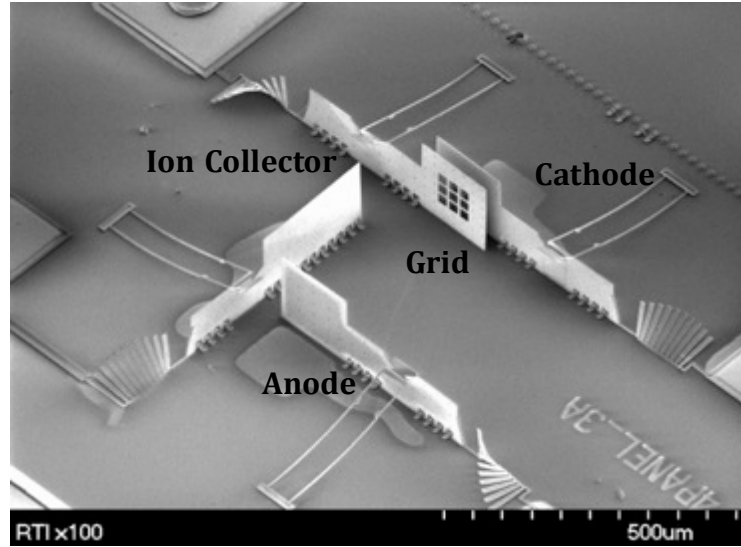


Figure 9: SEM Micrograph of MEMS Micro-ion Source

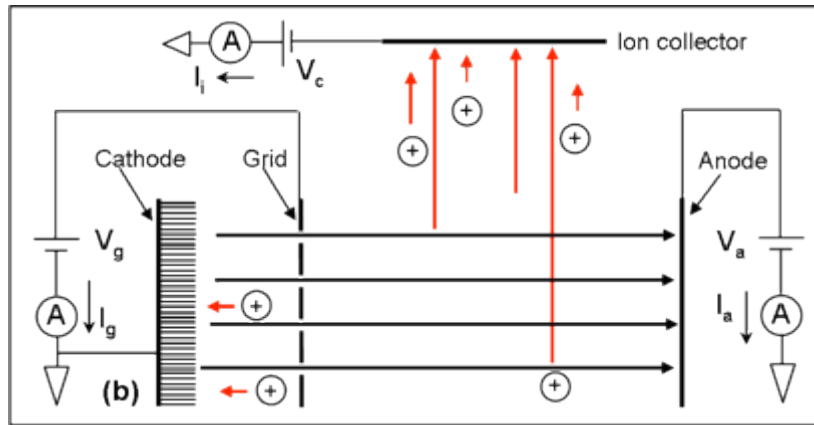


Figure 10: Functional Diagram of Micro-ion Source

2. Experiment and Results

We performed a triode characterization run on a micro ion source. The voltage on the cathode was held constant at -100V, the grid was stepped from 0 to 300V in 50V increments, and the anode was swept from 0 to 400V in 1 V increments. We measured the current on the anode at every combination of these voltages. The data is presented in Figure 11.

We notice that for low grid voltages, the anode current values remain in the nA range. This is expected as the diode is not field emitting for this range of grid voltages. When the grid voltage is greater than some threshold between 200 and 250V, the anode current increases with anode voltage. In this situation, the diode is field-emitting and electrons are accelerated through the micro ion source to the anode. However, we do notice that the current increase is inconsistent: because field emission is not a well controlled process, the field emission currents fluctuate a lot over time. This could explain why the anode currents exhibit spikes and troughs as anode current increases. We also note that at a grid voltage of 400V, the level of anode current is lower than it is at a grid voltage of 350V. This suggests that the grid voltage is positively charged

enough that it is attracting the field-emitted electrons back towards it once they pass through its holes. Applying the most extreme voltages to the panels is, therefore, not the solution to getting an appropriate distribution of electrons throughout the device. Nonetheless, this experiment shows that the anode is accelerating field-emitted electrons through the micro ion source. Future work will involve demonstrating that impact ionization is occurring.

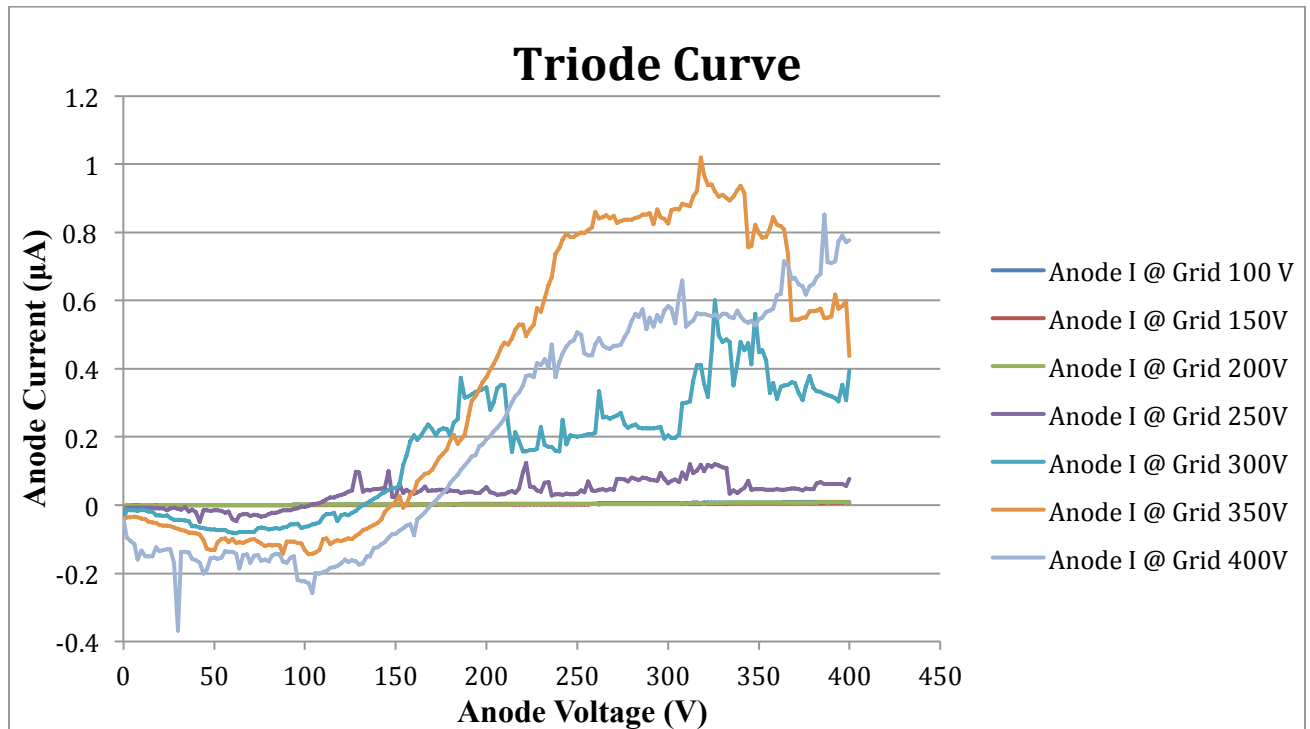


Figure 11: Triode Characterization Curves

3. LabVIEW VI triode_characterization.vi

This Virtual Instrument was built to run a triode characterization experiment. The user can select the cathode's voltage, the range of voltages the grid will step over, and the range of voltages the anode will sweep over. The grid's step and the anode's sweep can each be set to be linear or logarithmic. The experiment can be set to automatically run as many times as needed. The user also selects a name to give to the data run and can also input any notes before or while the experiment runs.

When the Virtual Instrument is launched, it communicates with the Keithley sourcemeters to set them up for sourcing voltage and measuring current, as seen in the discussion of diode_characterization.vi. The same low-level SCPI commands used in diode_characterization.vi are used to synchronize the Keithley measurements here as well.

As soon as the last sweep is finished, VI stores all the current measurements in a .txt file that bears the name given the data run. The eventual notes the user took are also placed at the top of the file. The rest of the file contains the measurements organized in a table in the following manner: each column represents an anode sweep for a certain grid voltage. The grid voltage values increase as you move towards the right in the file,

while the anode voltage values increase as you move towards the bottom of the file. This file is automatically stored in a folder bearing the name of the date of the experiment. The data is, therefore, stored and organized in a manner that is easy to access and process for future experiments on micro ion source prototypes.

5. Characterizing the Ion Beam

1. Ion Beam Detection

Because no micro ion sources were available to perform ionization tests on gases, a previously assembled macroscopic ion source was used to test how our ion beam imaging system functioned. The objective of this testing was to see if the ion beam detector could detect gases that were leaked into the chamber, ionized by the macroscopic ion source, and mass-separated in a magnetic field. Demonstrating this would confirm that our system is ready to characterize the ion beam from micro ion source prototypes once the next phase of testing begins.

2. LabVIEW VI camera_control.vi

After manually adjusting the power supplies that power the different parts of the custom mass spectrometer and leaking gases into the vacuum chamber, an ion beam is produced, mass-separated by a magnetic field, and its cross-section is captured by the beam imaging system described in 3.3 (see Figure 12). The Virtual Instrument camera_control.vi acquires and processes images from the Sony XCD-U100 that is programmed to continuously take pictures of the ion beam detector's phosphor screen. The gain, shutter, and brightness of the camera can be changed in real time. The stream of images is displayed in a window on the front panel of the VI on the computer. Using the computer mouse, the user can then draw a line through the cross-section in the display so that the program continuously calculates an intensity profile along that line and displays it in a graph on the screen as well. This enables the user to observe a mass spectrum in real time and the peaks that represent where ions are striking the detector. At any moment, the user can push a button to snap an image of the ion beam detector and the intensity profile along a line if one is drawn. All the files are placed in a folder bearing the name the user gives to the data so that they can easily found and processed later.

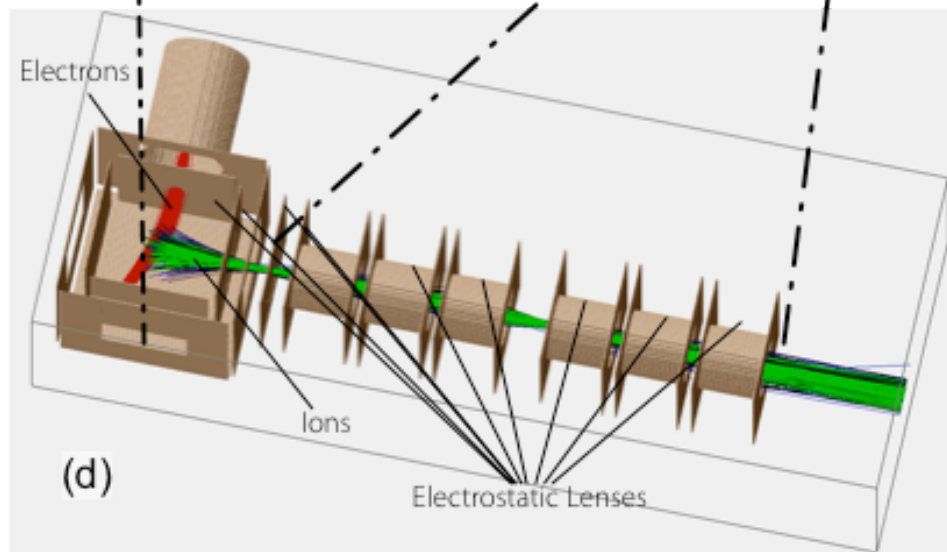
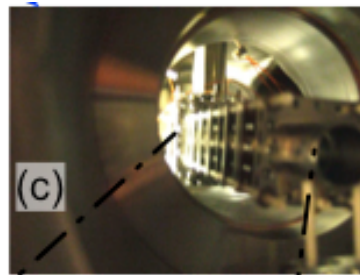
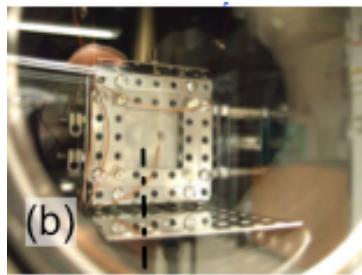
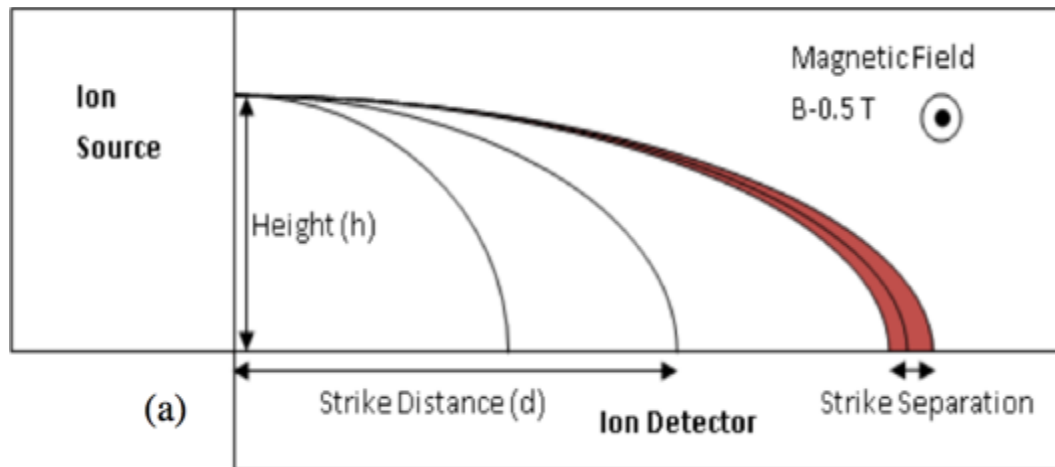


Figure 12: Ion Beam Detection with a Custom Mass Spectrometer

- a) Diagram of Mass-separated Ion Beam Detection
- b) Macroscopic Ion Source
- c) Electrostatic Lenses of Ion Source
- d) SIMION Simulation of Macroscopic Ion Source

3. Experiment and Results

We tested the beam imaging system by leaking nitrogen pressures of 4×10^{-5} Torr and 1×10^{-4} Torr into our vacuum chamber and using camera_control.vi to determine if our system could detect the mass-separated ion beam. By using the intensity profile feature of the VI, we acquired images and spectra expecting to see some intensity peaks emerge as the nitrogen pressure increased.

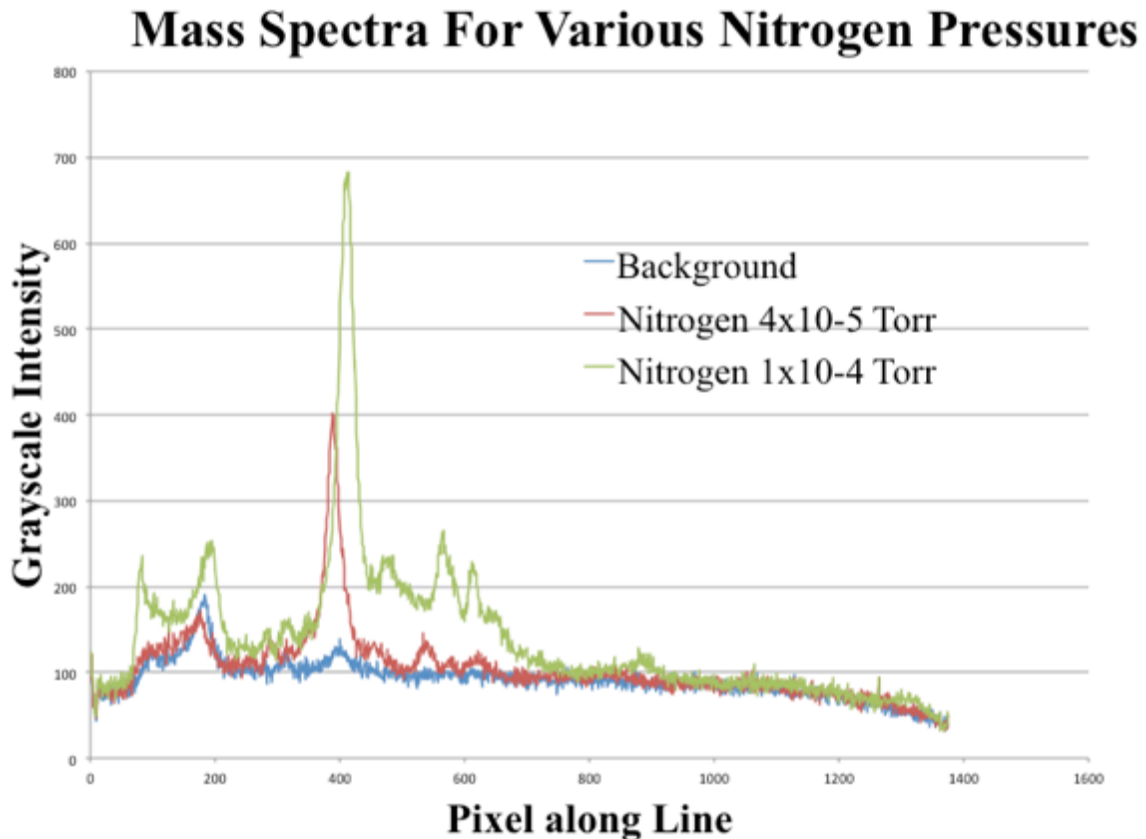


Figure 13: Mass Spectra for Various Nitrogen Pressures

The background spectrum exhibits some peaks, showing that the mass spectrometer is processing some of the background gases: water and nitrogen are commonly found in many rough vacuum systems as they make up a large proportion of air. The major peak of the background at pixel 190 represents ionized H_2O with an atomic mass unit (AMU) of 18, as our particular vacuum chamber has been known to have moisture issues. There is also a background peak at pixel 400, which we assume represent ionized N_2 with an AMU of 28.

As the nitrogen pressure increases, the general level of the background spectrum rises, but there are also peaks that rise significantly above the rest of the data. In particular, the peak at pixel 400 increases the most, confirming that that peak represents ionized N_2 . If we take the background nitrogen peak grayscale value to be about 100, then the increases in the nitrogen peak make quantitative sense too. By approximately doubling the nitrogen pressure from 4×10^{-5} Torr to 1×10^{-4} Torr, the grayscale value of the nitrogen peak effectively doubles from 300 to 600 if we subtract the background

value of 100. By observing the appearance and increase of peaks of light on the output of the ion beam detector, this experiment proves that our system is capable of detecting a mass separated ion beam.

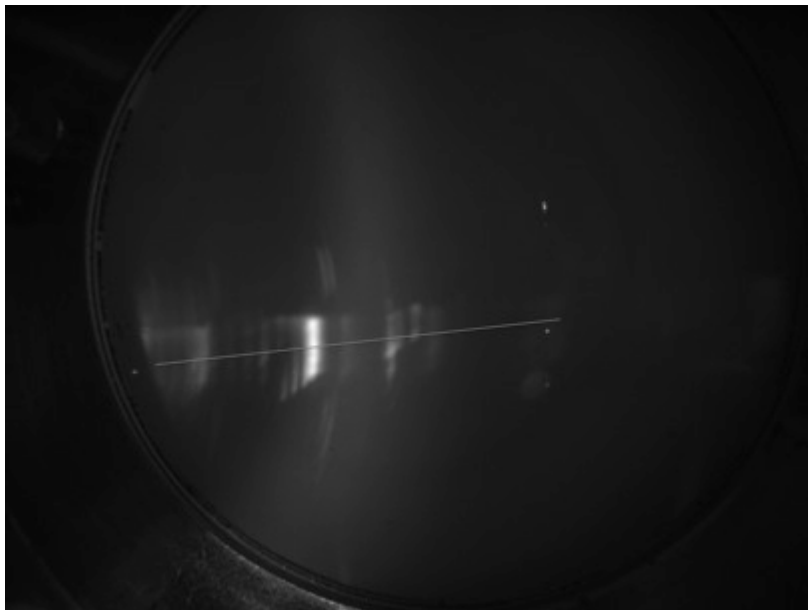


Figure 14: Image of Ion Beam Detector acquired by camera_control.vi for a Nitrogen Pressure of 10^{-4} Torr

(The grey line approximately represents the line used for spectra in Figure 13)

4. Improvements to LabVIEW VI camera_control.vi

However, the method of using intensity profile along a line produces noisy data. To reduce noise, the final version of the code takes advantage of the fact that the ion beam landing on the ion beam detector is two-dimensional, as seen in Figure 14. Instead of drawing a line through the ion beam, the user draws a rectangle enclosing the beam, like in Figure 15. The program then averages the pixels along one axis of the rectangle to eliminate noise and plots the values as a function of length along other axis.

Also, we noticed that when no experiment is performed, the intensity profile across the direction where ions land was not uniform but possessed a curved shape shown in Figure 16. We suspect that this is due to the non-linear lighting of the phosphor screen by the filament used for electron emission in the macroscopic ion source. To flatten the background curve, the user sets the system up to take an averaged intensity profile of a rectangular area when no ion beam is landing there as described in the paragraph above and in Figure 15. This background intensity profile will present the aforementioned curved features. By pressing a button on the front panel, the user indicates that the current spectrum represents the curved background and the VI will automatically subtract it from any subsequent reading. Comparing Figure 16 and Figure 17 shows that the flattening and averaging methods will provide a steady baseline for future characterization of ion beams.

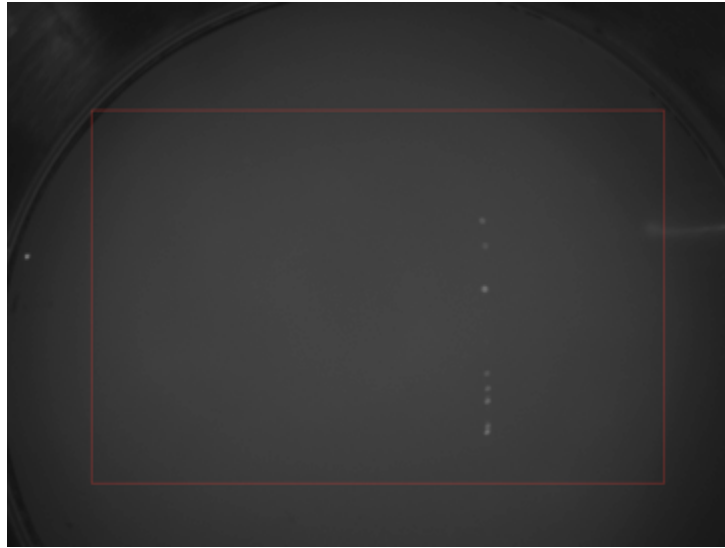


Figure 15: Picture of Ion-Beam Detector Phosphor Screen
 No Ion Beam was being fired at the system. Spots of light indicate cracks in the MCP

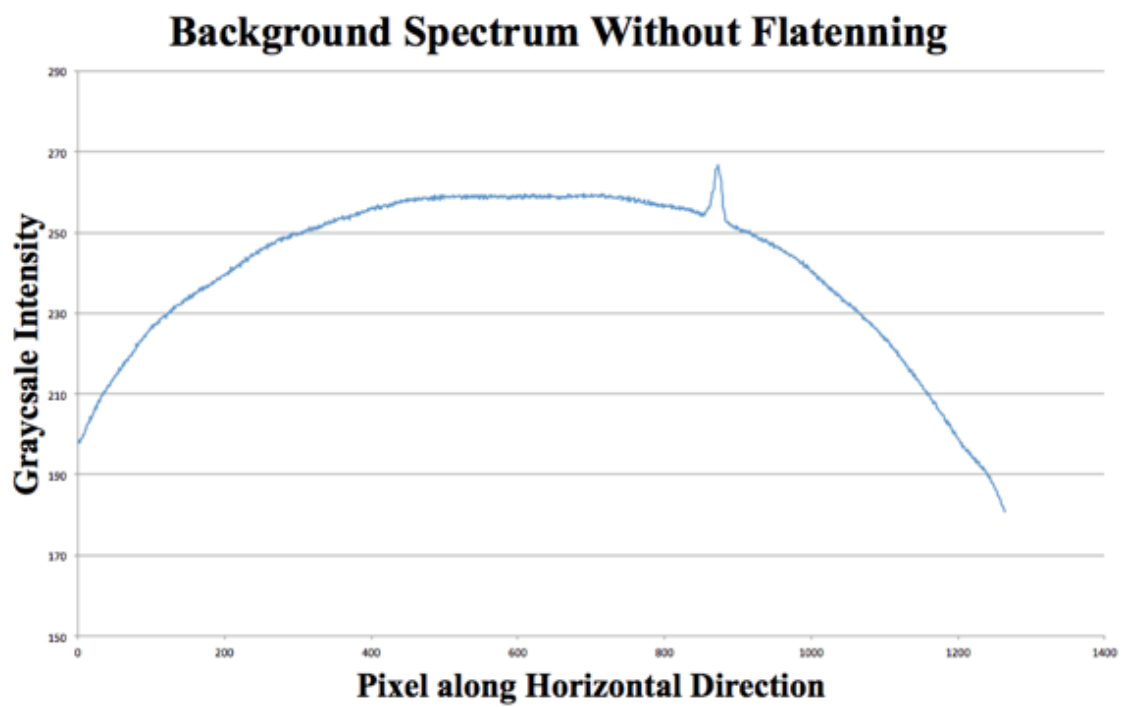
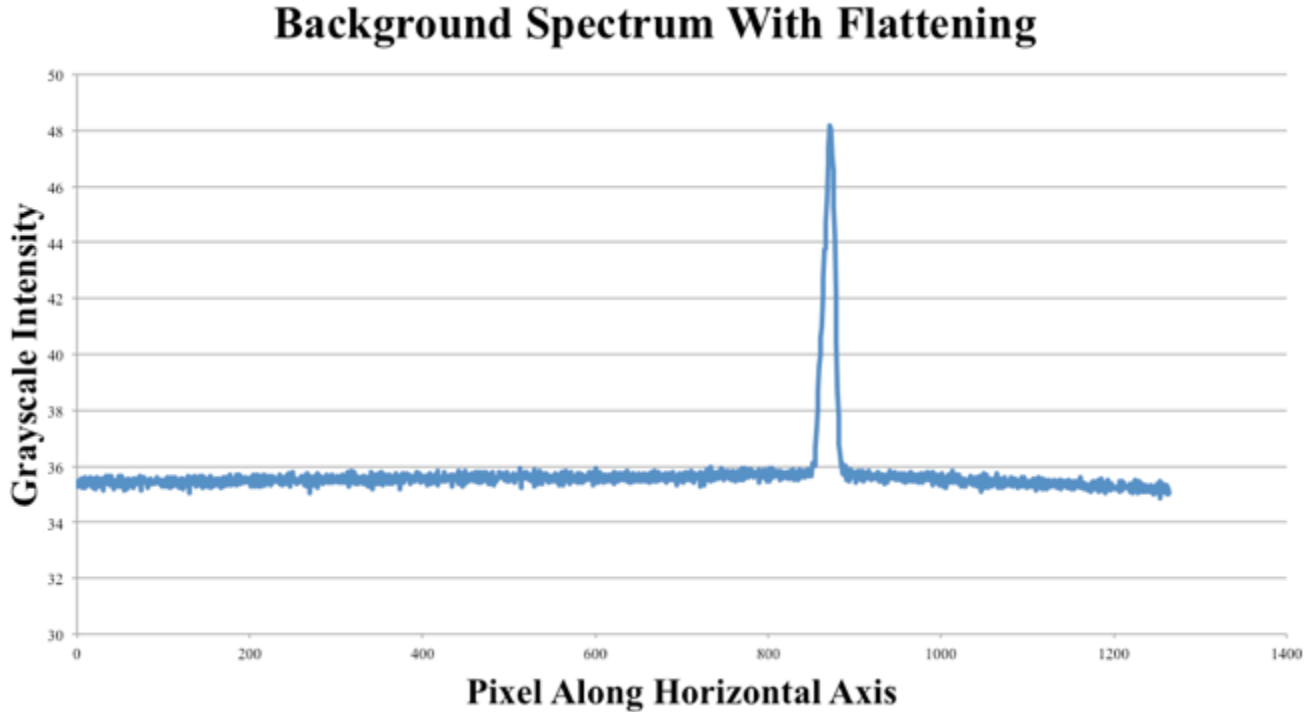


Figure 16: Background Spectrum without flattening
 (Computed from image in Figure 15)



**Figure 17: Background Spectrum with flattening
(Computed from image in Figure 15)**

6. Conclusion

We have developed hardware and software tools that characterize the different components of a microfabricated mass spectrometer. LabVIEW VIs `diode_characterization.vi` and `triode_characterization.vi` have been designed to perform diode and triode characterization tests on the microfabricated ion sources that make up the first essential component of the full device. These tools have demonstrated field emission of electrons in the diode and acceleration of these electrons through the triode for impact ionization. Though we could not work with ion beams produced by microdevices, we also proved that our automated beam imaging system can detect mass separation in an ion beam. By implementing real-time imaging of mass spectra, we used `camera_control.vi` to detect an increase in peaks of nitrogen. Thus, the beam imaging system has proven that it will be useful for future work on ion beams.

Future work will involve testing a new generation of micro ion sources and fully characterizing their properties, from field emission to impact ionization, with our software tools. We then hope to microfabricate Einzel lenses capable of producing an ion beam that can be mass separated in a magnetic field, and observing this separation with `camera_control.vi`. Long-term work also includes using coded aperture techniques to transform the ion beam into an encoded signal that will feature high intensity and high resolution to increase the spectroscopic ability of the microfabricated mass spectrometer.

Acknowledgements:

I would like to thank Dr. Jeffrey Glass, Dr. Charles Parker, Dr. Scott Wolter, and Zach Russell for all their help. I have benefited from their financial support, but most importantly I have gained knowledge, technical ability, and confidence from working them. I would especially like to thank Zach, whom I worked a lot with and who taught me a lot about vacuum, charged particle optics, and science.

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