Scanning Electron Microscope on a Chip

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1 Introduction

The scanning electron microscope (SEM) is an essential tool for a large part of the scientific community with applications ranging from archeology and geology, to biology and material science. Currently a standard SEM is about is about the size of a full-grown man [Figure 1]. The miniaturizing of an SEM would greatly expand the utility it provides and make it a more powerful instrument, as discussed in section 2.1.1. An SEM uses an extremely focused electron beam to create high-resolution images of up to 10,000x magnification [1]. The electron gun, which provides this beam, is the main obstacle to miniaturization. This project centers on creating a micron scale electron gun.

1.1 SEM Background

The SEM's breadth of utility comes from its versatility in imaging a large variety of samples over a wide range of magnifications. It can obtain three-dimensional images with a magnification between 10-10,000x, as well as elemental analysis and crystal structure [1]. The versatility of the SEM stems from its ability to detect a wide array of signals with secondary electrons, backscattered electrons, and characteristic x-rays being some of the most common. The SEM also benefits from the ability to image and characterize a diverse field of specimens ranging from the organic world of plants animals and insects, to inorganic materials such as crystals and polymers. The primary component of an SEM is a highly focused electron beam that is swept over the specimen in a raster pattern. From the electron interaction with the specimen various signals, including secondary electrons, characteristic x-rays, and backscattered elctrons, can be collected to characterize its surface topography, crystallography, and composition. Because of its high quality and tightly focused electron beam the SEM is an invaluable instrument in an extensive range of scientific fields.

1.2 SEM Miniaturization

An SEM's size, as well as the need for a high voltage power supply on the order of 10-20 kV, limits its operation to that of a standard laboratory setting. Making a small and mobile SEM would allow it to be used directly in the field wherever it is need. Other recent miniaturization processes can give insight into how shrinking could affect the utility of an SEM. The development of the computer is an excelent example of how miniaturization has greatly increased its use and application. From the ENIAC, the fist computer in 1946, to the Iphone, released in 2007, the size of computers have been reduced from the dimensions of a large room to a small handheld device in just over 50 years [Figure 2] [2]. With this miniaturization the computer has gone from being used only in a handful of places across the globe, to being an essential part to almost every aspect of the modern world. Another drastic miniaturization process has occurred in the field of microfluidics. In this field significant gains have been made in being able to control small amounts of fluids with micron sized pumps on a nano-meter level. A recently developed device at UCLA allows for 1,000 simultaneous chemical reactions to be carried out on a single palm-sized chip [Figure 3] [3]. If these same reactions were attempted 15 or even 10 years ago, they would require a large bench top set-up taking up thousands of times more space. By miniaturizing a SEM, similar results in the technology's use and utility can be expected.

Figure 1: a photo of a standard laboratory SEM. The electrical column is shown in the foreground and the control column in the background.

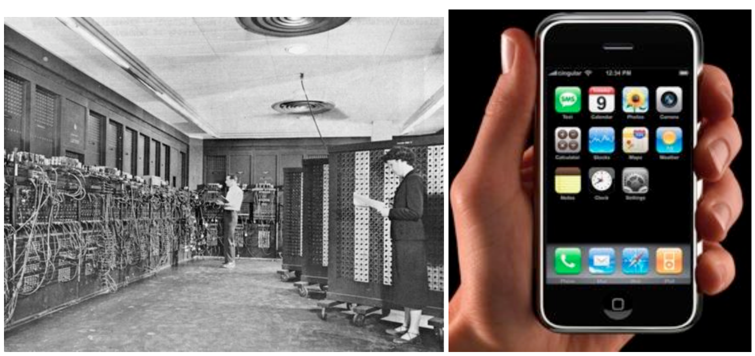

Figure 2: ENIAC first computer built in 1946 (left), Iphone developed in 2007(right).

Figure 3: Microfluidic device developed at UCLA capable of running 1,000 chemical reactions simultaneously.

1.3 The Electron Gun

In order to miniaturize the electron gun, an understanding of how electron emission takes place is required. There are three main types of emission: Photo, Thermionic, and Field [Figure 4**]** [4]. Photo emission occurs when photons with a sufficient energy are incident on a material. These photons excite the valence electrons to a high enough energy state that they can escape the potential barrier from the nucleus, resulting in electron emission. Similarly, thermionic emission occurs when a material is heated to a sufficiently high temperature. This raises the electrons' energy, causing them to escape the potential barrier. Finally, field emission occurs when a high electric field is applied to a material. This lowers the potential barrier the electrons "feel" from the nucleus, allowing them to tunnel out of the metal. This type of emission can be enhanced by creating a sharp tip that concentrates the electric field at that point, making the electrons feel a higher field than the external applied one [Figure 5] [5]. A more in depth discussion of field enhancement will be given in the theory section of this paper.

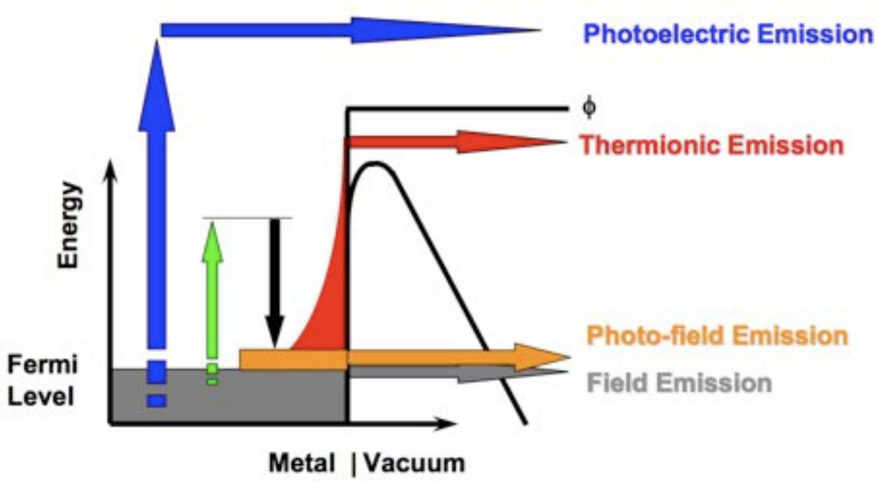

Figure 4: Overview of different electron emission mechanisms. Of importance field emission.

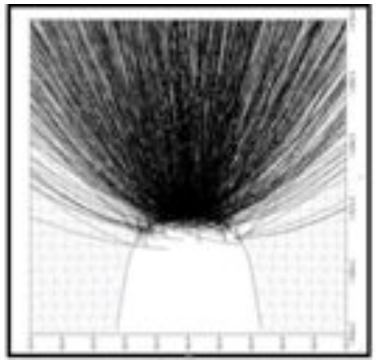

Figure 5: Graphical representation of electric field concentration at a sharp point.

1.4 Beam Quality

In designing the electron gun, consideration must be given as to which type of emission produces the best electron beam for an SEM. The two factors that determine the overall quality of the electron beam are current and brightness [1]. High current is important in order to achieve a good signal to noise ratio. If the current is too low the detector will not properly function, and will be unable to distinguish the image signal from the background noise. The second importance aspect is brightness. Brightness is a conserved quantity: the brightness achieved at the gun is the maximum brightness achievable at the specimen. The brightness at the specimen directly effects the resolution of the image because it influences the diameter of beam at the sample [1]. If the beam diameter is not small at the specimen, then the quality of the SEM image will be poor. The figure below shows some measurements for gun brightness and currents [Figure 6] [4]. A more complete discussion of brightness will be given in the theory section of the paper.

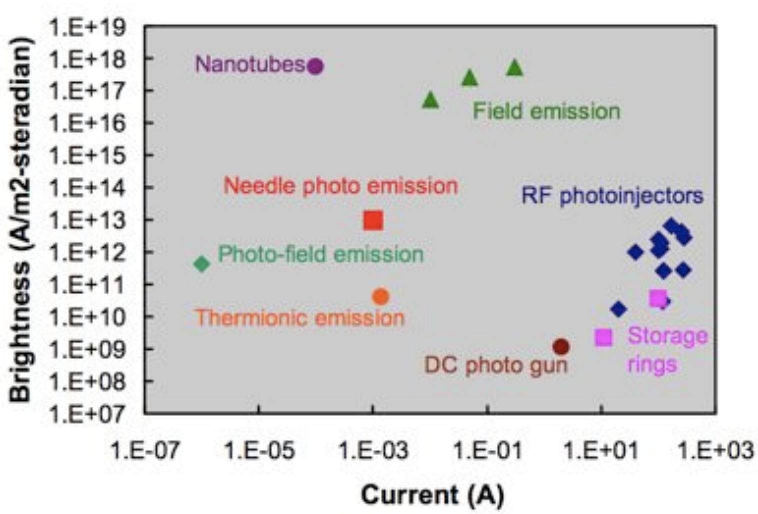

Figure 6: Graph of current versus brightness for various types of electron guns. Note that field emission provides highest combination of brightness and current.

To better understand the project, in the next section we describe the operation of the SEM in more detail. Following that, a section on the relevant theoretical models will be presented.

2 How an SEM Works

A SEM consists of two main components the electrical column (the primary technical apparatus of the machine) and the control console (concerned with producing an image from the electrical column). The focus of this paper will be primarily concerned with the electrical column, shown below [Figure 7] [6]. The electron gun provides the source of the imaging beam, which is generally created by applying a high voltage, 10-20 kV, between the gun and the anode. Next the beam is focused to a tiny spot on the specimen by the lens, which is on the order of 10 nm. This ability to create a very small spot on the sample is essential to producing a high-resolution image. Finally, the beam is moved over the specimen in a raster pattern by the electric scanning coils, which allows it to be swept over the entirety of the sample [1]. The final step to creating an image is to collect a number of different signals from the beam sample interaction. The most commonly collected type is secondary electron emission, and will be the main tool of the miniature SEM.

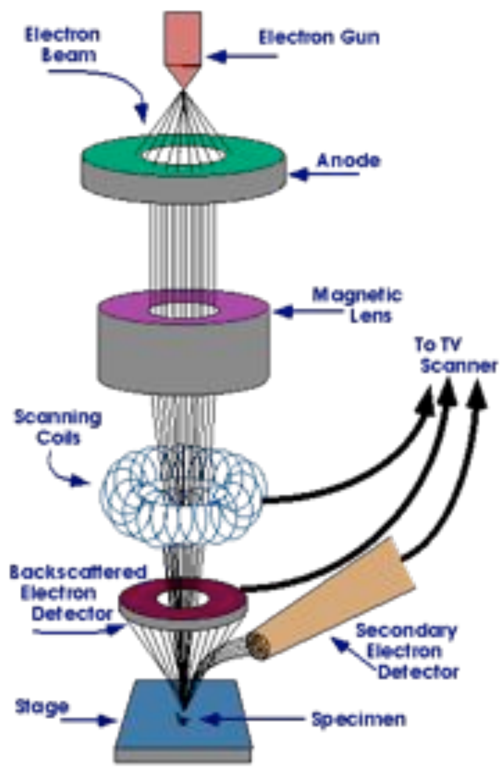

Figure 7: Components of an SEM. Of importance are: electron gun, magnetic lens, and canning coils.

2.1 SEM as Lab-on-a-chip

2.1.1 Benefits and Applications

To provide the greatest benefit from miniaturization this project is attempting to shrink down a SEM to a lab-on-a-chip scale. Having a lab-on-a-chip SEM will allow for rapid, mobile testing that can be done in large volume through an array type assembly. With a chip sized SEM testing can be done directly in the field without having to be taken back to the laboratory, saving time and resources. Additionally a lab-on-a-chip SEM should be significantly less costly than its larger laboratory sized counterpart. The possibility even exists for the device to be created as a inexpensive disposable instrument, adding the benefit of being useable almost anywhere in the world, especially in usually inaccessible rural or undeveloped areas.

2.1.2 Microfluidics

In order to shrink this technology down to a chip scale a conceptual leap will be made in how the beam is scanned. In a conventional SEM the beam is scanned electrostatically in the x (horizontal) and the y (vertical) directions to create a two-dimensional plane over the specimen. However, when the SEM is condensed to fit on the flat plane of a chip, the beam can only be scanned in the one dimension (parallel to the plane of the chip). The second perpendicular plan will be provided by a microfluidic pump, which will move the sample at a right angle to the plane of the chip [Figure 8]. This technology is advantageous to the lab-on-a-chip design because of recent advancements made in microfluidics. Specifically, this technology allows for nano-meter scale control over specimens in microfluidic devices, as demonstrated by J. Wang of UCSD [7]. Also these pumps allow for tiny volumes of liquid to be precisely managed, in applications such as insulin delivery in diabetic patients [Figure 9] [8]. This type of accurate and precise movement control should work well as the second dimension required for the raster pattered scan, and fits perfectly the size restrictions need to fit on a chip.

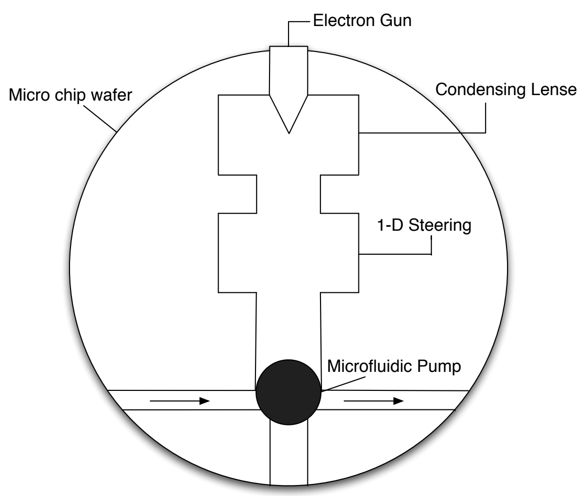

Figure 8: Prototype drawing of a lab-on-a-chip SEM. Microfluidic pump is perpendicular to the plane of the chip.

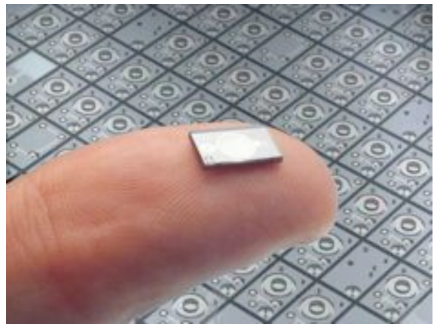

Figure 9: Microfluidic pump to deliver insulin to diabetic patients .

2.2 Shrinking SEM Components

Creating a lab-on-a-chip SEM requires that all the steering, focusing, and detector components be miniaturized to the micron scale. The next paragraphs will give a brief discussion on how this can be done using existing technologies and production techniques.

2.2.1 Focusing

The magnetic lens focuses the electron beam onto the specimen so that the spot size is at its minimum value there. This is often done using an Einzel lens [Figure 10]. An Einzel lens consists of three or more cylindrical or rectangular wires aligned in series. The middle wires are set to a variable potential depending on the desired focal length. With this setup the lens concentrates the electrons down to a point as shown below [Figure 11] [9]. Miniaturizing these component can be easily accomplished, as all it requires is the fabrication of micron scale electrical wires constructed in cylindrical or rectangular shapes. The industry of computer processing currently uses similar process that could easily be adapted to fit the application of the Einzel lens.

Figure 10: A perspective look at a typical Einzel lens

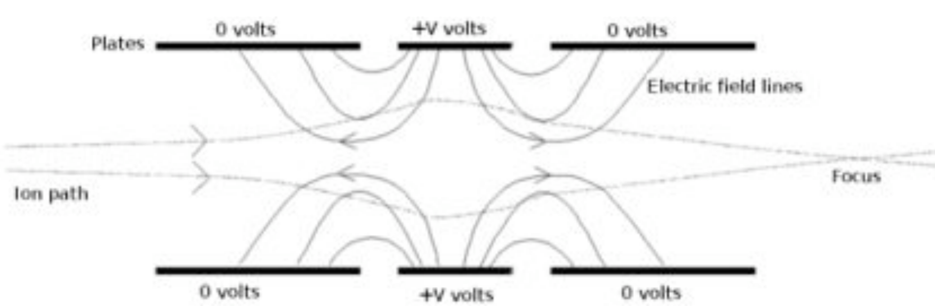

Figure 11: Einzel lens showing the path of ions passing through it. Six plates are parallel to the ions path with the middle set to a particular voltage depending on the desired focal length.

2.2.2 Steering

Electrical steering in a SEM is accomplished by scanning coils or deflection plates [Figure 7]. The voltage of these plates is varied depending on the energy of the electrons as well as the distance they are being deflected. Miniaturizing the SEM will require these plates to be shrunk down to the micron scale, meaning a higher voltage will be needed to achieve the same deflection. However, the samples use in the lab-on-a-chip SEM will be smaller and require less deflection of the beam. This should roughly cancel out the increase in voltage caused by the smaller plates. The steering, like the focusing components, should be supported by current techniques used by the micro processer industry.

2.2.3 Detector

Creation of a micro detector should be supported by current micro machining process [Figure 12] [1]. However, if creating a detector becomes an obstacle it can be easily remedied by simply having a small handheld detector that plugs into the chip. The chip would then be the disposable apparatus of the SEM, while the plug in detector would still afford the advantages of being small and mobile. The secondary electron detector must be able to detect electrons in 10-30 keV range as well as have a photomultiplier gain of 10^5-10^6 times [1]. The Everhart-Thornley detector is almost universally used in today's SEM, and a similar concept would likely be use in the lab-on-a-chip version.

Figure 12: Small electron detector, which could potentially plug into lab-on-a-chip SEM.

3 Theory

3.1 Introduction

This section will introduce and review the basic scientific theories behind the micro electron emitter. First a discussion of field generation by the pyroelectric crystal will be given. This will be followed by an overview of the tip field enhancement. Lastly, a more thorough analysis of the electron beam quality and brightness will be give.

3.2 Field Generation

In field enhanced emission a high voltage is applied to the gun typically on the order of 10-20 kV. In the case of a mobile lab-on-a-chip SEM it is not practical to have this high voltage power supply. To overcome this obstacle pyroelectric crystals can be used both as the high voltage and emitter source. Pyroelectric crystals generate high electric fields when subjected to a temperature gradient. This temperature gradient causes the crystal to become electrically polarized with the positive and negative sides depending on whether the crystal is being heated or cooled. The electric field generated by the crystal is given by the following equation:

$$E_0 = \frac{-\gamma \delta T}{\varepsilon_0 d_{cr} + \varepsilon_{cr} d_g} d_{cr} ,$$

where E_0 is the electrical field, γ the pyroelectric coefficient of the crystal, δT the change in temperature during which emission occurred, ε_0 the vacuum permittivity, the crystal permittivity, d_{cr} the thickness of the crystal, and d_g the distance between the emitter and the metal target [10]. The main point of interest in this equation is that the distance to the anode, d_g, is the most easily varied parameter and greatly affects the magnitude of the generated field.

3.3 Needle Field Enhancement

Needle field enhancement essentially concentrates the electric field lines down to a sharp point, resulting in multiplying effect of the field felt there [Figure 5]. The magnitude of this enhancement is given by the following equation:

$$E_{tip} = E_0 \frac{a^2}{b^2} \frac{\eta^3}{\arctan(\eta) - \eta} ,$$

where a is the needle length, b is the radius at the base, and $\eta = \sqrt{1 - b^2/a^2}$ [11]. Field enchantment is generally on the order of 100x with being $a \approx 10 \mu\text{m}$ and $b \approx 1 \mu\text{m}$. The type of material used as well as the technique used to create the tip limits achievable sharpness of the tip, and as such the field enhancement. Also of importance to note is that the emission current from the tip decreases as the area of the tip gets smaller, i.e. the field enhancement must be balanced against the needed current.

Brightness is a measure of the electron beams spread in transverse space as well as phase space. This factor is the overall most important quality of an electron beam, because it cannot be enhanced past the value it starts with at the gun. The formal definition of brightness is given by the equation:

$$B_n = \frac{1}{4\pi^2} \frac{I}{\varepsilon_{rms}^2} ,$$

where I is the current, and ε_{rms} is the root mean square of the emittance [1]. Emittance can be better understood as the six dimensional spread in phase space of the electron shown in the figure below [Figure 13] [4]. What makes brightness such an important factor in an SEM is that it is directly related to the resolution of the image. In order to create a high-resolution image, the diameter of the beam must be very small. This allows for small details to be resolved on the surface of the specimen. If the beam diameter is too large then these details will be missed. The beam diameter is roughly related to brightness by the following: $\text{Diameter}_B \propto \frac{1}{\sqrt{B_n}}$ [1]. This means that the higher the brightness the tighter the beam will be and the higher the resolution of the image.

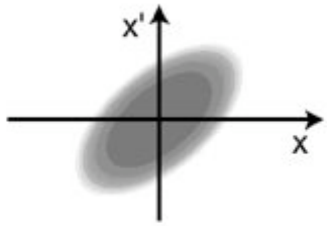

Figure 13: graphical representation of an electron's spread in phase space.

4 Design and Materials

As previously mentioned the electron gun will be made from a pyroelectric crystal. Past studies of these materials by Rosenman have shown that these crystals produce high enough electric fields to induce electron emission [10]. There exist several different pyroelectric materials some of which are listed in the table below [Table 1]. The highest pyroelectric coefficients are likely the best materials for the electron gun because they will produce the highest electric field. However, all the examined materials had roughly equal coefficients, and ease of availability dictated the use of lithium niobate.

Table 1: Table of different pyroelectric coefficients

Material	Lithium Tantalate	Lithium Niobate	TGS
Pyroelectric coef. $C/(cm^2K)$	2.3×10^{-8}	0.82×10^{-8}	2.3×10^{-8}

In order to achieve emission the applied field from the pyroelectric crystals must be enhanced using the needle tips previously discussed. For this a variety of wedge and cone shaped micro emitters were designed to assess how different geometries affected the guns performance [Error! Reference source not found.9].

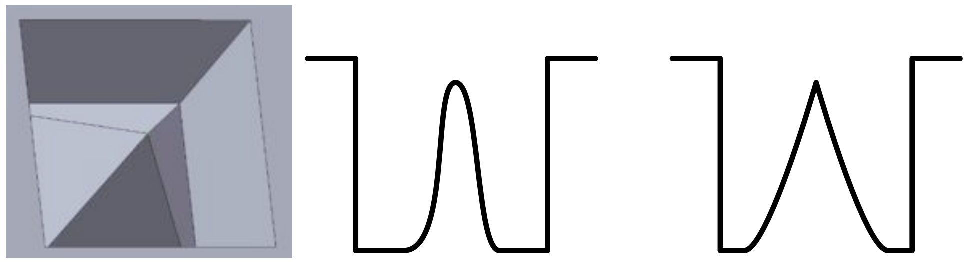

Figure 14: Drawing of tip emitters. Pyramidal design (right), two-dimensional view of cone design (middle), and two-decisional view if wedge design (right).

5 Fabrication

To construct the micro emitters lithium niobate was gold coated. This was done to ensure an even distribution of charge over the surface and to prevent emission from sources other than the emitters. The tips were then micro machined into the crystals using a focused ion beam. Arrays of slightly varied emitters where then made using wedge and cone shaped templates [Figure 15, and Figure 16]

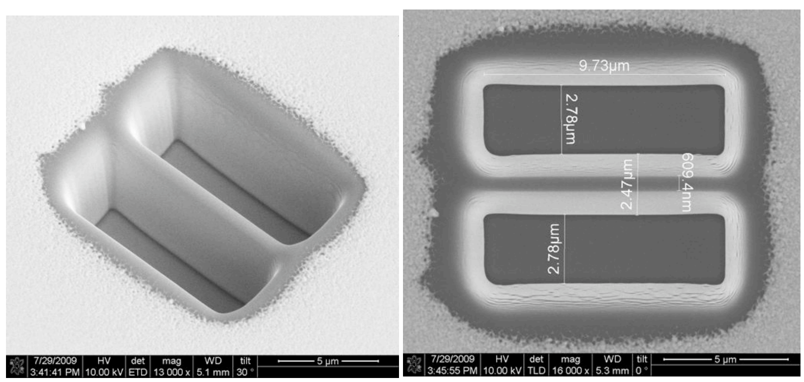

Figure 15: SEM image of a wedge shaped micro emitter. Perspective view (left) and overhead view (right).

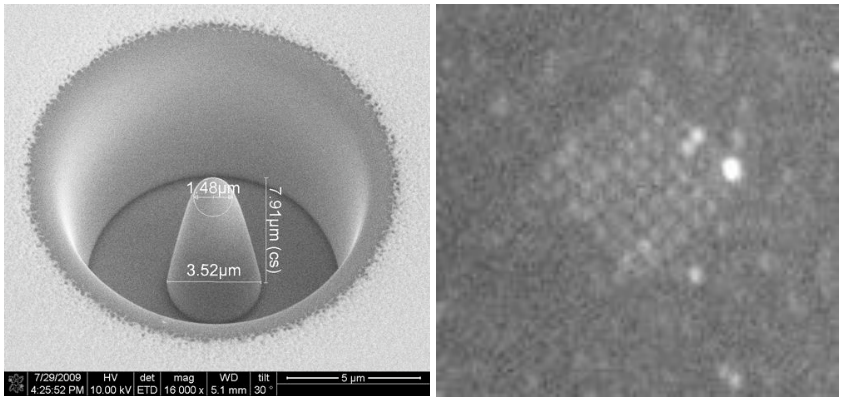

Figure 16: SEM image of a cone shaped micro emitter (left). Image of 8 by 8-square array of micro machined emitters.

6 Experimental Set up

Once the crystals emitters were machined they were placed onto a variable voltage heating apparatus shown in [Figure 17]. The heating rate was controlled through a variac power supply. The temperature was monitored by a thermister that was placed in a copper sheet on top of the heater. A scintillator was then placed in front of the crystal so that any electron emission could be detected. Then all components were placed within a vacuum box that allowed viewing through a window placed in from of a camera [Figure 18, and Figure 19]. The whole chamber was then pumped down to $1\times10^{-6}\,\mathrm{Torr}$ before heating cycles were started.

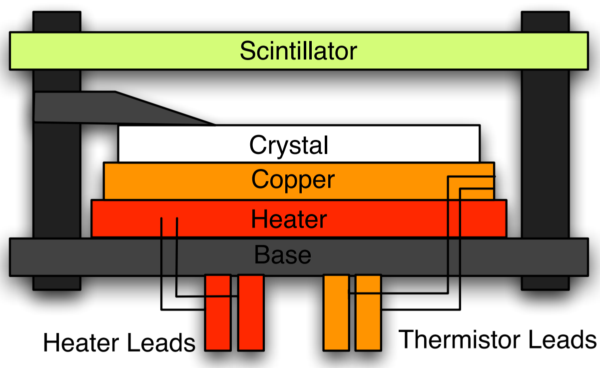

Figure 17: Drawing of the heating apparatus for the pyroelectric crystal. Heater is attached to a variable voltage source to control heating rate.

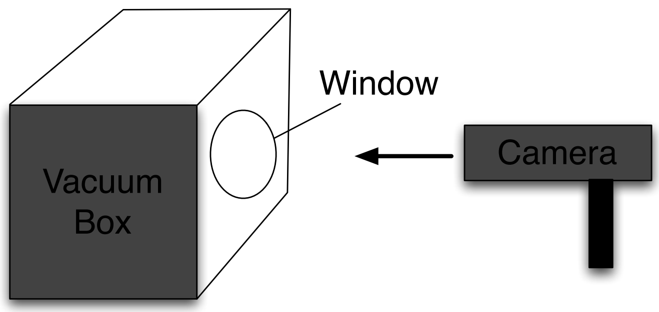

Figure 18: Heating apparatus is inserted into the vacuum box, which can then be viewed through the window with a camera.

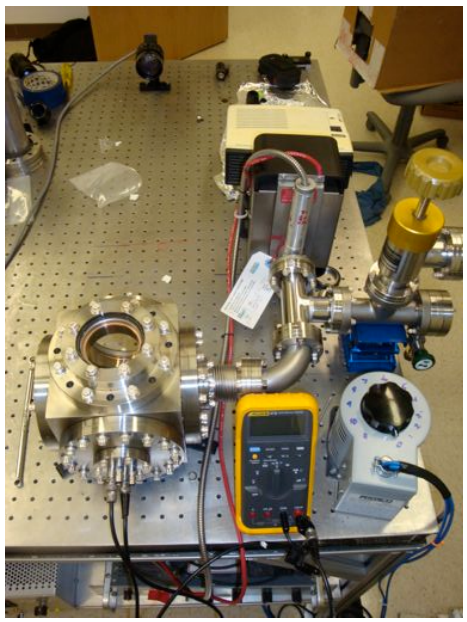

Figure 19: Bench top set up for experiment. Vacuum box is located in the lower left Conner. Pumping apparatus is on right.

7 Results and Analysis

When the crystals were heated periodic flashes lasting less then one second were observed. These flashes were semi random both in frequency and in location. The fact the flashes occurred in several different places can be attributed to the cracks that formed in the crystal while under the stress of heating and cooling. This created many places the crystal could emit from besides the machined emitters. No continuous emission was seen from the crystal.

The reason no continuous emission was seen from the crystal was likely due to insufficient field strength at the emitters. This discrepancy in field strength resulted from an ambiguous anode distance. Originally the intended anode was the gold coating over the crystal. This would have produced a field strength of $E_g = 3.67\times10^7\,\text{V/m}$. However, it appears that the gold did not act as the anode, probably because it was at the same potential as the rest of the crystal. Instead the anode was likely the metal in the vacuum chamber about 10 cm away. This caused the produced field to fall to $E_g = 7.75 \times 10^3 \text{V/m}$, well below emission range. It is also likely that the field enhancement of the emitters was not high enough for emission. In order to achieve emission for most metals the field at the tip must be between 3-10 GV/m. To achieve this field strength enhancement must be on the order of 100x. However, the machined emitters only had a field enhancement of about 22x. Much greater field enhancement is possible, and has be demonstrated on many other needle sources [4]. Though the technique chosen for this experiment may need to be changed in order to achieve this.

Though continuous emission was not observed in this experiment, valuable insight was gained into understanding the sensitivity of the material to changes in geometry, especial in positioning of the anode. Further work must be done to minimize the anode spacing, which could be accomplished by putting some sort of insolating coat between the crystal and the gold. Additionally optimizing the field enhancement will require further refinement in using the focused ion beam. Once these immediate steps have been taken. Characterization of the electron beam must be done in terms of the beams brightness and current output.

8 Future Work

Currently machined emitters have been observed to emit for over durations of over two minutes. However this emission was not seen in the experiment of this paper, and not yet understood geometric changes have prevented reproducibility of the continuous emission. In moving forward with this work a better understanding of how the positioning of the anode affects the generated field must be done using simulations. With that knowledge construction of a micro emitter with the necessary field generation and field enhancement can be made. Once the geometry of the experiment is fully understood and continuous emission are seen, major work is needed in characterizing the quality of the electron beam. This includes adapting existing instruments used to measure beam current and emittance to fit our specific experiment design. Once characterization of the beam is complete, engineering of the actual lab-on-a-chip SEM can begin. It is conceivable that an operating SEM could be produced within 3-5 years.

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