Miniaturized Environmental Scanning Electron Microscope for In Situ Planetary Studies

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ABSTRACT

The exploration of remote planetary surfaces calls for the advancement of low power, highly-miniaturized instrumentation. Instruments of this nature that are capable of multiple types of analyses will prove to be particularly useful as we prepare for human return to the moon, and as we continue to explore increasingly remote locations in our Solar System. To this end, our group has been developing a miniaturized Environmental-Scanning Electron Microscope (mESEM) capable of remote investigations of mineralogical samples through in-situ topographical and chemical analysis on a fine scale. The functioning of an SEM is well known: an electron beam is focused to nanometer-scale onto a given sample where resulting emissions such as backscattered and secondary electrons, X-rays, and visible light are registered. Raster scanning the primary electron beam across the sample then gives a fine-scale image of the surface topography (texture), crystalline structure and orientation, with accompanying elemental composition.

The flexibility in the types of measurements the mESEM is capable of, makes it ideally suited for a variety of applications. The mESEM is appropriate for use on multiple planetary surfaces, and for a variety of mission goals (from science to non-destructive analysis to ISRU). We will identify potential applications and range of potential uses related to planetary exploration.

Over the past few of years we have initiated fabrication and testing of a proof-of-concept assembly, consisting of a cold-field-emission electron gun and custom high-

voltage power supply, electrostatic electron-beam focusing column, and scanning-imaging electronics plus backscatter detector. Current project status will be discussed.

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INTRODUCTION

Mankind has always been fascinated with the heavenly bodies that comprise our Solar System. We have made it a point to explore and understand these bodies in an attempt to satisfy a collective curiosity and to understand the origin and evolution of the Solar System. Whether it is the moon, Mars, or other planetary body that we wish to explore, we rely on suitable instrumentation to deliver the information needed.

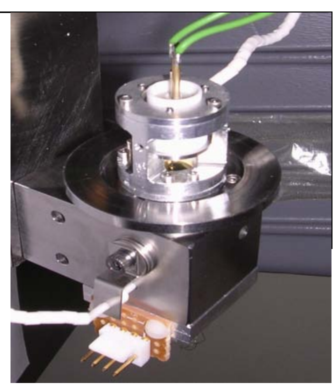
With a focus on functionality, low-power, miniaturization our group has initiated development of a miniaturized Environmental Scanning Electron Microscope (mESEM) capable of nanometer-scale resolution, *in-situ* topographical imaging and compositional x-ray fluorescence mapping of uncoated natural and synthetic samples. The diversity of measurements that can be made with a SEM (and particularly one with environmental mode capabilities) makes it an ideal instrument for a variety of planetary missions. This is highlighted by the fact that there has been multiple previous mini-SEM developments intended for various missions.

The concept of a miniaturized SEM is a not new one. The SEM and Particle Analyzer (SEMPA), developed in the late 1980's specifically for the Comet Rendezvous Asteroid Flyby satellite (since canceled), achieved ~40nm resolution, operated on a relatively low power of 22W, and weighed roughly 12kg (Conley et al. 1983; Albee & Bradley 1987). Utilizing current technologies, our novel design will permit an even smaller, lighter, lower-power version that is easily adaptable to a variety of missions. Additional efforts in this field have produced a range of results concerning the development of a mini-SEM or miniature electron focusing column (Khursheed 1998; Gross 2005; Callas 1999; Roberts et al. 1997; Yabushita et al. 2007).

For the sake of simplicity, we have limited our development to a lunar version of the mESEM. While many of the components are directly translatable to other environments - such as Mars - other components will need some modification (i.e. vacuum system). The lunar mESEM instrument goals are summarized in Table 1.

Table 1. Below is a list of the desired mESEM characteristics. The Overall system dimensions include all support electronics. The power estimate is based on our current high-voltage power supply design.

Parameters	***Goals***
Electron Gun/Column/Scanning Assembly	
Size	2" long, < 0.5" diameter
Mass	< 50g
Imaging Resolution	<100nm
Maximum Accelerating Voltage	10kV



Maximum Field of View	5-10mm square
Sample Preparation	Minimal – No Coatings
Overall System Dimensions	< 8"x4"x4"
Total Power	15W

Our efforts to date have resulted in proof-of-concept mESEM components (electrostatic-electron focusing column and scanning system) from which we have obtained our first focused image (Fig. 1). We have recently made progress on the remaining proof-of-concept components which include a custom electron gun and associated power supply system, and complete scanning/imaging system plus backscatter detector. Our final instrument will also include an Energy Dispersive Spectrometer (custom Silicon Drift Detector), which is outside of the scope of this current effort.

This work is a collaborative effort between NASA MSFC, Advanced Research Systems, the University of Alabama Huntsville, the University of Tennessee Knoxville, Case Western Reserve University, with contributions from Johns Hopkins Applied Physics Laboratory.

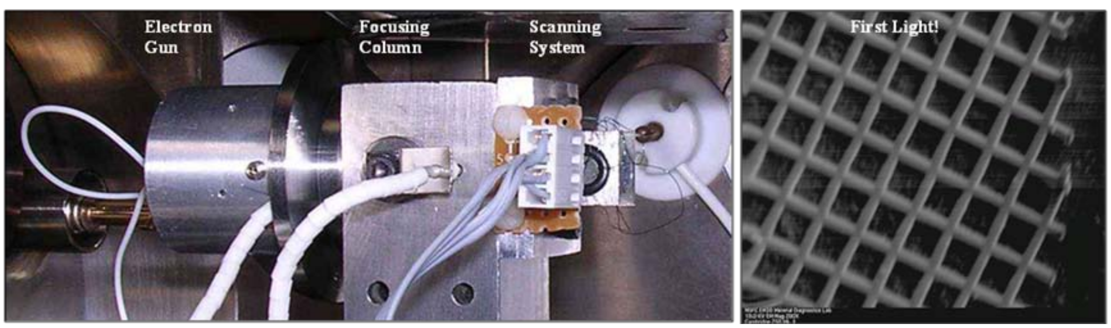


Figure 1: The image on the left shows three proof-of-concept mESEM components that have been fabricated and tested. The resulting imaging resolution (of a few microns) is much larger than that of the final version mESEM, due to the relatively large emission region that results from using a thermionic electron gun compared to that of a FEG (for which our focusing column was designed).

LUNAR SURFACE SCIENCE

The return of humans to the Moon is a major step in NASA's plan for exploration. It is imperative that we prepare for the science and engineering challenges that will be an intrinsic part of this vision, especially as it pertains to the study of lunar regolith.

Although considerable research has already been performed on lunar regolith collected during the Apollo Missions, this sampling represents less than ten percent of the lunar surface (Heiken et al. 1991; Jolliff et al. 2006). The successful realization of a lunar outpost will require significant expansion of our knowledge relating to lunar regolith (pertinent to In Situ Resource Utilization (Chambers et al. 1994; Chambers et al. 1995). The mESEM's ability to permit in situ morphological and chemical characterization of lunar regolith will minimize the need for sample return and allow for the differentiation of unique samples tagged for Earth return.

Morphological and chemical characterization of lunar regolith in laboratories on Earth has been routinely accomplished using SEMs and Energy Dispersive x-ray Spectroscopy (EDS) (McKay et al. 1991). The utility of the mESEM will be discussed relative to its ability to determine mineral and glass compositions, particle size distribution and morphologies, types of rocklets (~1-10mm rock chips) and their mineralogies, and general characteristics of lunar regolith components. These data relate to the science of soil formation and its utilization for resources (e.g., production of oxygen (Taylor & Carrier 1992; Taylor & Carrier 1993) from both the Moon's Maria and Highland terrains.

Further reading on the benefits of a lunar mini-SEM can be found in our sister paper by Thaisen et al., 2010 of these Proceedings.

STUDIES OF EXPOSED MATERIALS

In addition to science studies, the mESEM would be a powerful tool for studying the deleterious effects of the lunar environment. High-resolution images can capture surface corrosion, micrometeorite impact effects, UV-induced material degradation, and the effects of stresses imposed by the extremes of temperatures present on the lunar surface. These effects can show up as a loss of thermal, electrical and optical properties and diminished structural integrity. The mESEM would allow the analysis of fracture surfaces to identify common failure modes such as overload, fatigue, and creep (McEvily 2001). Coincident chemical analysis would allow for identification of residues and reaction products formed during lunar operations. For long-term human habitation, such studies are vital to ensure selection of appropriate materials.

It is worth noting that these studies are especially important in the harsh Martian surface environment. The mESEM would be ideal for carrying out contamination and corrosion studies; and similar studies of how manufactured materials react or degrade on the surface of Mars – essential if we are ever to have a human presence there. The Martian environment is 1) highly corrosive due to the presence of a strong oxidant capable of rapid destruction of organic compounds; 2) highly abrasive due to suspended, micron-scale dust driven at high velocities by seasonal dust-storms; 3) highly charged due to static voltages generated by the movement of this dust in an effectively nonconductive atmosphere; and 4) photochemically active because of the unfiltered high UV flux. The mESEM provides a means to directly and empirically study corrosive processes, in situ, and their associated risks through the examination of purposefully exposed materials.

METHODOLOGY

A standard SEM takes up the space of a large desk, weighs about half a ton, and requires kilowatts of power to operate. The transformation of an SEM to a planetary exploration instrument necessitates considerable miniaturization. Our approach is a complete rethinking of the basic SEM design that has prevailed for over 60 years, and makes use of a combination of innovative materials and construction techniques which have been validated in our prototype. The nature of the lunar environment (i.e. high ambient vacuum of ~10^{-12} Torr) allows for simplification of the overall instrument design that can then be modified for use on Mars.

The main components of an SEM include: an electron gun; electron focusing lenses; a deflection/scanning system; sample chamber or interface, electron and x-ray detectors; and vacuum system. Electrons generated in the gun propagate through the electron-optics assembly (consisting of precisely placed apertures and an electrostatic lens) and are focused onto the sample. The scanning coils raster this focused beam across the sample to create the subsequent image and characteristic x-rays. We have started development of proof-of-concept components including: a custom electron cold-Field-Emission (FE) gun, focusing column (with electrostatic lens system), and scanning/magnification system. Using our electron focusing column and scanning system combined with an off-the-shelf thermionic electron gun and support electronics from a commercial SEM, we were able to obtain our first focused image of a copper grid standard (Fig. 1).

Because we used a thermionic cathode for our initial electron-focusing column testing, the resulting imaging resolution did not meet our goal of <100nm. Since this time, we have been able to successfully fabricate and test a cold FEG and control system, for which our column was designed to be used with.

FIELD EMISSION ELECTRON GUN

The electron gun being developed is a cold field emitter that utilizes an off-the-shelf Hitachi tungsten cathode. A Butler-like triode configuration is employed, and consists of a field emitter tip followed by a first- and second- anode (Butler 1966). A large applied field between the field emitter tip and the first anode causes electrons to tunnel out of the tip (Gomer 1961). These electrons are then accelerated towards the second-anode, which is typically at ground. The accelerating voltage of the gun is defined as the voltage between the field emitter tip (i.e. cathode tip) and the second-anode. The extraction voltage is the potential difference between the cathode tip and first anode. One can use the Fowler-Nordheim equation (Fowler & Nordheim 1928), in modified form (Murphey & Good 1956) and restated in more general terms by Forbes 1999, to describe the relationship between emission-current density, J, and local field at the surface, F:

$$J = \lambda\, a\, \phi^{-1} F^{2} \exp(-\, \mu\, b\, \phi^{3/2} F^{-1}),$$

$$a = e^{3} / 8\pi h = 1.541 \times 10^{-6} \text{ A eV V}^{-2},$$
$$b = 4/3\, (2m_e)^{1/2} / e\nabla = 6.830 \times 10^{9} \text{ eV}^{-3/2} \text{ V m}^{-1},$$

where λ and μ are correction factors, a and b are universal constants, and ϕ is the work function of the emitter, e is elementary charge, h is Planck's constant, $\nabla = h/2\pi$, and m_e is the mass of an electron. A small increase in the extraction voltage (related to F) will result in a large increase in the total emission current. If the potential difference between the cathode and first-anode becomes too large, the fine cathode tip can be damaged. Precise control of the extraction voltage is required to avoid this and to maintain emission current stability.

MECHANICAL CONFIGURATION

The previous versions of our FEG mechanical design resulted in undesired results due to unexpected breakdown in the cathode chamber and high-voltage power supplies. As such, we have since designed and fabricated a new, more robust version that will be refined (further miniaturized) once the control system is proven.

Key features of this electron gun include:

- Fine horizontal alignment mechanism for centering the cathode tip to first anode aperture.
- Vertical alignment mechanism, accomplished via a finely threaded, graduated fitting which allows the field emitter tip to be placed at the desired distance from the First Anode, to within ~10μm.
- Alignment of the tip to the first anode is verified under an optical microscope before the second anode is attached.
- Ceramic spacers are used to electrically isolate the tungsten cathode from the First anode and to isolate the second anode from the first.

The first anode is an off-the-shelf platinum Hitachi aperture and the second anode is a small aperture drilled directly into a stainless steel cap. A Faraday cup is attached directly behind the second anode to measure the current at that point. Figure 2 illustrates this design.

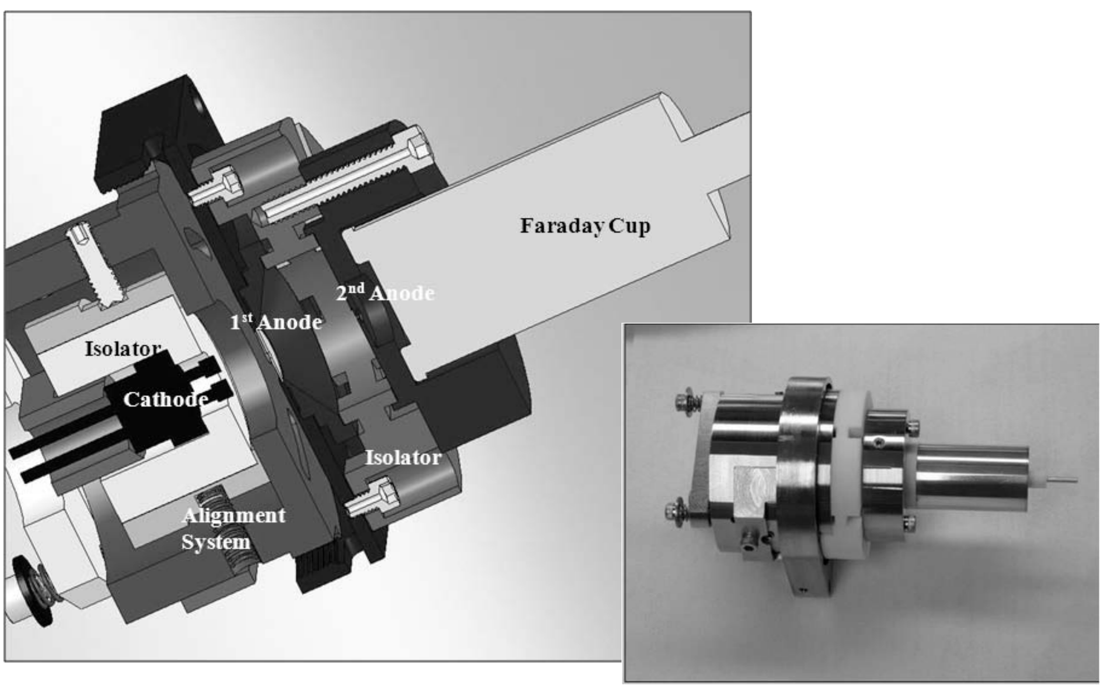


Figure 2. This diagram depicts the main components for our electron gun. The image on the bottom-right corner is the assembled gun, with Faraday cup attached just after the second anode.

EMISSION CURRENT CONTROL

A high-voltage power supply (HVPS) assembly and control system for the electron gun have been developed by collaborators at the University of Alabama Huntsville and with input from additional team members. The electron gun operates at a maximum accelerating voltage of 10kV.

Main Components/Features include:

- Three compact off-the-shelf switching power converters procured from Ultravolt, Inc. Two of these generate the voltages on the cathode and first anode, and the third functions as an isolator, to allow the first anode power supply to be ground referenced to the cathode high voltage.
- Power for the high-voltage supplies is provided by a commercial laboratory supply for testing. This supply can be easily replaced by a battery or alternate portable source at a later time (total power to run these three supplies is ~10 W).
- The entire HVPS system (without any repackaging of the Ultravolt units or electronics boards) fits easily inside a small enclosure (~8” x 4” x 4”). Further miniaturization is possible with custom designed power supplies and/or repackaging of these supplies and electronics.
- Control & monitoring for the power supply configuration are currently accomplished with the use of potentiometers and multimeters. Eventually, we will use microcontrollers. The user dials-in the desired emission current and accelerating voltage, and the potential on the first anode is automatically adjusted to maintain the desired current. Maximum accelerating voltage is 10kV.
- A flashing circuit has been developed to allow us to keep the cathode tip atomically clean.

TESTING & DISCUSSION

The electron gun has, thus far, only been tested with a blunted Hitachi tungsten FE cathode, vertically aligned to within 50μm of the first anode. The entire gun assembly sits inside of a large spherical vacuum chamber (capable of operating at high 10^{-10} Torr). Figure 3 shows SEM images of a sharpened tip versus a blunted one. The blunted tip – created by ripping the tip off by placing it in a high electrical field – has a much larger tip radius than the sharpened one. The effect of this is a diminished total emission current. These field emitters are relatively expensive and easily damaged – especially in the presence of high-voltage breakdown in the gun or even in the high-voltage supplies themselves. This preliminary testing has allowed us to insure the safety of all instrument components and of the high-voltage assembly itself (as parts of it are floating at 10kV).

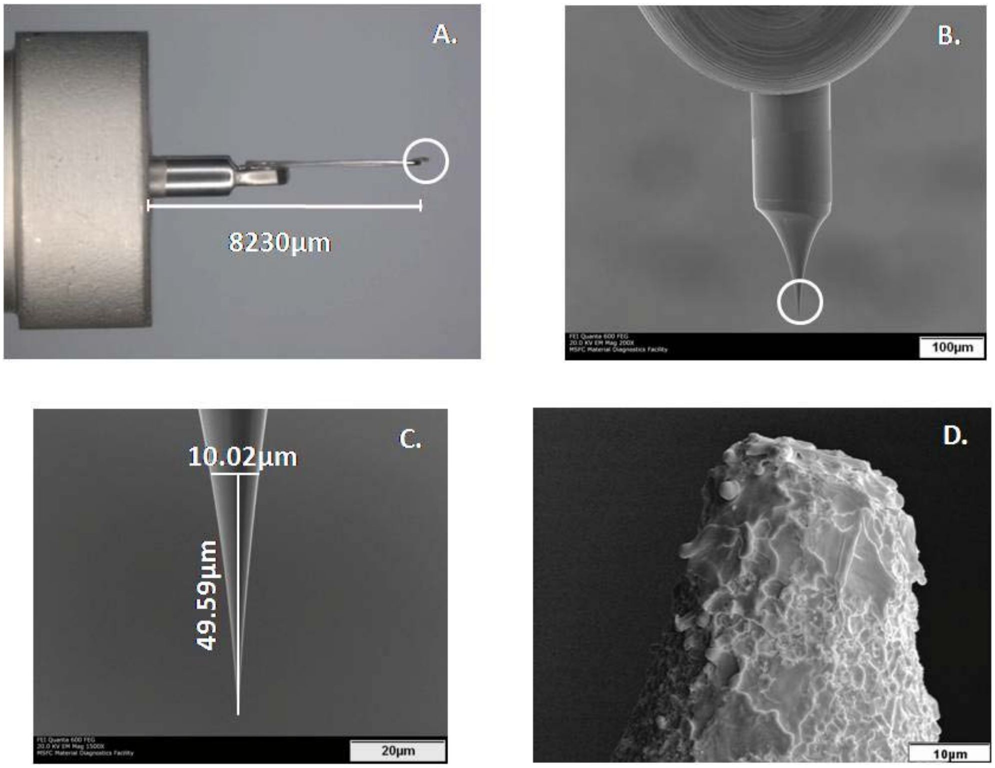


Figure 3. These images were taken using a commercial FEI Quanta 600 FEG SEM and depict the FE cathode at various magnifications. Image A) shows the complete tungsten cathode structure; B) is a magnification of the circled region in Image A; C) is a magnification of the circled region in Image B., and clearly shows the sharpened tip structure. Image D) is of a blunted tip structure (rotated 180^{o}). Images are courtesy of G. Jerman (NASA MSFC).

With this blunted tip installed in our electron gun we were able to successfully and repeatedly regulate emission current from the cathode. Table 2 shows results from three input currents, I_{in}. The current on the second anode surface, I_{a2}, was recorded along with the current seen by the Faraday cup, I_F and the extraction voltage, V_e (i.e. the voltage between the cathode and first anode). The accelerating voltage was 10.13kV and at a chamber pressure of $1x10^{-8}$ Torr.

Table 2. Three trials showing the input current to the cathode (I_{in}), extraction voltage applied to maintain this set current (V_e), and resulting currents monitored on the second anode (I_{a2}) and in the Faraday cup (I_F).

Parameter	Trial 1	Trial 2	Trial 3
I_{in} (μA)	0.52	1.0	2.4
I_{a2} (μA)	0.22	0.46	1.12 – 1.2
I_F (nA)	0.4	0.8	2
V_e (kV)	3.44	3.59	3.78 - 3.88

The ranges in the Trial 3 are due to the fact that emission was not stable. This is likely due to the fact that we were using a blunted tip rather than a sharp one. The data that is reported here gives reports approximate values, as these trials were intended for system check-out before a sharpened tip was installed in the electron gun. Further, these results are somewhat polluted due to leakage current between the regulation circuit and power supplies.

One can see from Table 2 that the extraction voltage increases accordingly as the input current is increased. It is also evident, yet not surprising, that the majority of the current produced in the cathode goes onto the first and second anode surfaces, rather than into the Faraday cup. To successfully image and perform EDS, one typically needs only a beam current of a few to 10nm (e.g. Erdman et *al*. 2009). We expect that with a sharpened tip installed, much more of the current will make it through the second anode, than seen in the above trials.

In parallel with these tests, we are conducting simulations on the electron gun configuration to determine the presence and effects of aberrations. These models will be used for optimization of the next iteration. The program we are using for this is Charged Particle Optics software from Scientific Instrument Services, Inc.

SUMMARY

Progress of the mESEM development is such that we have successfully fabricated and separately tested our electron-focusing column (electrostatic lens), obtaining our first focused image. We have also fabricated and tested an electron-gun assembly with a blunted Hitachi cathode and were able to successfully regulate the desired beam current using our high-voltage power supply system. For the next round of testing we will insert a sharpened cathode tip into our gun assembly and fully characterize the gun operation in terms of total beam current. Following the successful completion of this testing, we will mate our column/scanning system with the gun assembly for further characterization.

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