A MINIATURIZED VARIABLE PRESSURE SCANNING ELECTRON MICROSCOPE (MVP-SEM) FOR IN-SITU MARS SURFACE SAMPLE ANALYSIS.

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Introduction: The Miniaturized Variable Pressure Scanning Electron Microscope (MVP-SEM) project, funded by the NASA Planetary Instrument Concepts for the Advancement of Solar System Observations (PICASSO) Research Opportunities in Space and Earth Sciences (ROSES), will build upon previous miniaturized SEM designs [1, 2] and recent advancements in variable pressure SEM's [e.g., 3] to design and build a SEM to complete analyses of samples on the surface of Mars using the atmosphere as an imaging medium. This project is a collaboration between NASA Marshall Space Flight Center (MSFC), the Jet Propulsion Laboratory (JPL), electron gun and optics manufacturer Applied Physics Technologies, and small vacuum system manufacturer Creare. Dr. Ralph Harvey and environmental SEM (ESEM) inventor Dr. Gerry Danilatos serve as advisors to the team.

Variable pressure SEMs allow for fine (nm-scale) resolution imaging and micron-scale chemical study of materials without sample preparation (e.g., carbon or gold coating). Charging of a sample is reduced or eliminated by the gas surrounding the sample. It is this property of ESEMs that make them ideal for locations where sample preparation is not yet feasible, such as the surface of Mars. In addition, the lack of sample preparation needed here will simplify the sample acquisition process and allow caching of the samples for future complementary payload use.

Science Requirements: Science requirements were needed to assist in defining the components and capabilities of the MVP-SEM hardware, thus providing engineering constraints. The following science requirements were identified for the MVP-SEM:

Science Requirement 1. The MVP-SEM shall have an imaging system capable of resolving uncoated objects or phases 100nm in size or better (adjustable magnification to 5000X or greater), with a minimum magnification of 20X. This requirement provides an imaging capability with a sufficiently wide field of view to observe relatively large features in a sample, as well as an imaging capability with a greater resolution than any microscope flown to Mars to date. This requirement indicates the need for an ESEM, as the samples will be uncoated and unable to mitigate charging without a surrounding gas.

Science Requirement 2. The MVP-SEM shall be capable of determining the geochemistry of multiple uncoated samples; three or greater mineral types to a precision of 2 weight percent per major element. A quick look at the geochemistry of a mineral or phase using energy dispersive spectroscopy (EDS) is a major reason why geologists use SEM technology. Quantitative EDS allows identification and initial characterization of a mineral. This requirement puts additional constraints on the hardware and software of the MVP-SEM. The electron beam must be strong enough, and the environmental distance minimized sufficiently, to yield ample X-rays for a quantitative EDS analysis. The sample chamber will contain standards to assist in calibration of the instrument's EDS measurements, allowing any interference from the gas to be removed from the signal.

Science Requirement 3. The MVP-SEM shall be capable of analyzing materials with little to no sample preparation. The current focus of the instrument is sedimentary petrology; samples are expected to be removed from the surface of Mars and placed into containers on a sample wheel for analysis without significant preparation (i.e., no other preparation but sieving). This sample wheel would be capable of placing a sample within a chamber of controlled pressure for SEM analysis. The team is currently focusing on a sample chamber in which the vacuum levels are well-controlled in order to design a system that sufficiently addresses the imaging and EDS requirements. The rationale for a closed sample chamber at present, opposed to an open chamber the landed vehicle can place on the martian surface, is discussed further in the environmental testing portion of this abstract.

Science Requirement 4. The MVP-SEM shall complete analyses on multiple samples. This requirement does not define a maximum number of samples or analyses. Currently, the team is looking at a sample wheel that can eject some samples and cache others for either collecting for return to Earth, or providing analysis of a sample prior to a destructive analysis in a complementary payload.

Science Requirement 5. The MVP-SEM shall produce images capable of being downlinked to Earth. This requirement places data handling constraints on the images, and thus the required pixels in the detector, as well as software needed to process the images before they are sent to Earth.

Science Requirement 6. The MVP-SEM shall operate on a Mars-landed spacecraft. Not only does this requirement encompass all launch and payload requirements, it provides the constraints on the sample chamber gas.

Environmental Testing: In order to define the optimum operating parameters for the MVP-SEM, a study is underway at MSFC using a FEI Quanta 600 Field Emission Gun SEM. An analog gas with the composition CO_2: 95.49%, N_2: 2.68%, Ar: 1.62%, O_2: 0.13%, CO: 0.08 is used in the chamber to mimic the martian atmosphere.

Secondary electron imaging was the first detection method studied. Each setting listed here was experimentally varied to determine the optimum imaging conditions, as well as the conditions at which imaging was no longer possible: electron gun voltage (5-15kV), sample chamber pressure (0.5-7.5 Torr), magnification (20X to 20,000X), beam current (3 apertures).

Two imaging standards were utilized; the first with diamonds 400nm wide with 300nm spaces between, the second with dots 200nm wide with 100nm spaces between dots. These standards provided the necessary resolution measures for the MVP-SEM system to be designed to meet Science Requirement 1.

Two detectors were used; in the low vacuum mode, a Large Field Detector located to the side of the sample, and a Gaseous Secondary Electron Detector coaxial to a 500µm Pressure Limiting Aperture (PLA) for the ESEM mode. The environmental distance (ED, between the bottom of the PLA and the sample) was varied and recorded. Spot size was set at 3.9.

Digital micrographs were collected with the following parameters: 3.67 megapixel size, 2048 by 1792 pixels, image aspect ratio 8 by 7, and 20µs electron beam dwell time per pixel. In the 5000X magnification images, pixel size was calculated to be a 13.7nm square. A 100nm feature with this resolution would be represented within a 7 by 7 pixel matrix, or approximately 50 pixels.

Image quality was categorized into three tiers: 1) Good – the available image contrast is sufficient to clearly define both coarse and fine features (Figure 1, left), 2) Marginal – image contrast is restricted and barely sufficient to clearly define both coarse and fine features (Figure 1, center), and 3) Unusable – image contrast is severely restricted and insufficient for defining fine features and barely sufficient at defining coarse features (Figure 1, right).

With the large field detector, at beam voltages of 12 and 15kV, and ED 1.3mm, image quality at all magnifications was good up to 7.5 Torr. At 10kV and ED 1.3mm, image quality began degrading at 200X and 7.5 Torr. At 5kV and ED 1.3mm, image quality decreased at 3.5 Torr and was unusable at and above 5 Torr. When the ED was increased to 6.3mm, images were unusable at all beam voltages and at pressures of 5 Torr and above. At 5kV and ED 6.3mm, imaging is unusable at 2 Torr and above. At 10kV and ED 6.3mm, imaging is unusable at 3.5 Torr and above. At 12kV and ED 6.3mm, imaging degrades at 3.5 Torr and is unusable above 5 Torr. At 15kV and ED 6.3mm, imaging degrades at 5 Torr.

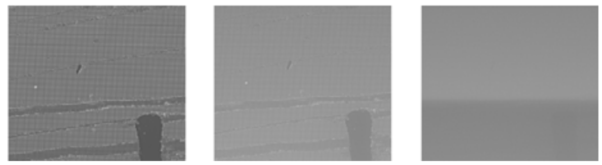

Figure 1: "Good" image (left), "Marginal" image (center), and "Unusable" image (right).

With the gaseous secondary electron detector, at 15kV, imaging is good up to 6 Torr at ED 1mm and 2mm; at ED 4mm, imaging degrades at 6 Torr; at ED 6mm, imaging degrades at 4 Torr. At 10kV, imaging degrades at 6 Torr at ED 2mm; at ED 4mm, imaging degrades at 4 Torr; at ED 6mm, imaging degrades at 2 Torr. At 5kV, imaging degrades at 6 Torr at ED 1mm; at ED 2mm, imaging degrades at 4 Torr; at ED 4mm, imaging degrades at 2 Torr; at ED 6mm, imaging degrades at 1 Torr.

The tests show that at 15kV, and a pressure of 5 Torr (roughly Mars atmospheric pressure), the operational maximum ED for good imaging is approximately 3mm. At 10kV, the ED drops to 2mm, and at 5kV, 1mm. If an ED larger than 3mm is needed, the sample chamber pressure would have to be regulated. Later EDS testing may confirm the need for pressure regulation in the sample chamber. Thus, the team is already anticipating the sample chamber design will include pressure regulation.

An additional gaseous detection device, not available through FEI, will be constructed and tested at JPL in fiscal year (FY) 16. Detector geometry and size of the PLA will be modeled in FY16 as well; it is hoped the geometry can be modified to maximize the electron and X-ray counts to broaden the boundaries placed on sample chamber pressure.

What would you study on Mars with the MVP-SEM? Tell us anonymously at:

https://www.surveymonkey.com/r/VBNZNDZ

References: [1] Gaskin J. A. et al. (2012) *IEEE Aerospace*, doi: 10.1109/AERO.2012.6187064. [2] Thaisen et al. (2009) LPS XL, Abstract #1697. [3] Fitzek H. et al. (2015) *J. Microscopy*, doi: 10.1111/jmi.12347.