

MINIATURIZED SCANNING ELECTRON MICROSCOPE FOR THE MOON: ENERGY DISPERSIVE X-RAY SPECTROSCOPY STUDIES

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Introduction: NASA's renewed interest in returning humans to the moon has prompted the development of compact, low-power, multi-purposed instrumentation to support future missions (whether they will involve a lander, rover or more permanent structure). Recent discoveries of water made by NASA's Moon Mineralogy Mapper instrument on the Chandrayaan-1 orbiter [1], later confirmed by the Lunar Crater Observation and Sensing Satellite [2], have only acted to strengthen the already compelling case for a return trip.

Over the past few years, NASA Marshall Space Flight Center and colleagues have been developing a highly miniaturized Scanning Electron Microscope (mSEM) with chemical analysis capability via Energy Dispersive Spectroscopy (EDS). This powerful combination is particularly useful because it allows one to analyze the surface structure and composition of a sample simultaneously, giving insight into its formation and evolution, and for non-destructively assessing, *in-situ*, the effects of space weathering on materials.

Typically, SEM with EDS is not used as the primary instrument of choice for performing chemical analysis of geological samples. Rather, Electron Probe Micro-Analysis (EPMA) with Wavelength Dispersive Spectroscopy (WDS) capability is used. However, this requires somewhat complex sample preparation (flat, polished sections). WDS requires the use of several analytical crystals which adds complexity, weight, and size to the detector setup. As such, our group has chosen to develop an SEM with EDS. Advantages of using an SEM includes, but is not limited to, minimal sample preparation (if operated under suitable conditions) and ultra-fine imaging resolution. EDS requires only a source, an appropriate detector, and minimal sample preparation. In the case of the mSEM, the source is the incident electron beam (also used for imaging the sample surface), and the X-ray detector will be a single silicon-drift detector attached to readout electronics and solid-state cooling system.

Project Overview: We are interested in understanding limitations on the science that can be done with a SEM equipped with EDS before we develop an instrument that will operate remotely on the lunar surface. There are two methods of EDS commonly used with regard to an SEM: mapping EDS and point EDS. In the former, an X-ray map is created as the electron beam is scanned across the sample surface and typically requires longer electron-beam dwell times. In the latter case, an X-ray spectrum is

created at a single point on the sample. With our colleagues at the University of Tennessee in Knoxville (UTK), we have performed several point EDS experiments with a commercially available SEM, using terrestrial lunar-analog samples. The goal of these tests was to quantify the surface X-ray emission region from a given sample to understand the minimum interaction volume resulting from the electron-beam interfacing with a sample, for a given set of mSEM conditions (e.g., incident electron energy, sample type and size).

These results will allow us to understand the limitations that our mSEM on the surface of the Moon may present [3]. Specifically, by determining the smallest interaction volume between the incident electron beam and a lunar-, or terrestrial-analog sample, for a given incident electron beam energy (i.e., the mSEM accelerating voltage), we will be able to determine the smallest grain size that we can analyze without contamination from surrounding material. Depending on the composition of the sample, we may be able to adjust the accelerating voltage to further limit the size of the interaction volume.

We expect that the majority of our samples will be lunar soil (240-1000 μm) [4] and dust (<20 μm) [5], with a peak particle-size distribution for lunar dust collected in the Apollo 11 mission being ~100-200 nm [5, 6]. Larger grain-size samples will often contain constituents of the sample such as agglutinates, with its melted glass, or be covered by dust particles that adhere to the sample due to electrostatics [7]. In addition, 50-80% of dust sized particles are glass created by meteoritic bombardments which contain nanophase Fe^0 particles that are typically 3-30 nm [8]. If we determine that the smallest interaction volume for a particular mineral has an X-ray emission area on the order of 10 μm , yet the sample or region of interest on the sample is much smaller than this, then we will know that surrounding materials will be present in our spectrum.

To effectively quantify the smallest particle size, we need to be able to predict the area of X-ray emissions resulting from an interaction between a lunar sample and a focused beam of energetic electrons produced. This requires the development of models to study the interactions between the sample and incident electron beam and resulting X-ray emission. We are developing an algorithm to do this, and we have completed preliminary experiments for verification.

Algorithm Development: In a typical Field-Emission SEM the imaging resolution, which is dictated by the size of the electron beam at the sample,

is on the order of 10nm or better [9]. During point EDS analysis, the resulting X-ray emission originates from a much larger area than this, as interactions take place within the sample. This emission region is dependent on the size of the electron beam at the sample, the incident electron beam energy, and the sample composition. To determine the resulting emission region for a given sample, we are developing a Monte Carlo simulation. Once this algorithm is completed, we will compare our simulation results to existing programs such as Casino [10] and GEANT4 [11].

Experimental Testing: We will compare results from our simulation to experimental testing of terrestrial analog samples. For these tests, we used a Quanta 600 FEG Field Emission Environmental SEM from FEI. In an attempt to mimic the functional characteristics of the mSEM we set a maximum accelerating voltage of 10kV. Our colleagues at UTK provided three samples: Olivine (-dunite), Plagioclase, and Pyroxene. Each was ground to give a distribution of grain sizes ranging from several microns to a few hundred nanometers. A standard SEM sample stud that was covered with two-sided carbon tape held the samples in place. Spot EDS was performed on various grains and the resulting spectrum was analyzed. If the spectrum included a carbon peak, then the assumption was that the area of emission was larger than the grain size. Figure 1 is an image of a single olivine grain that is $\sim 7.3 \mu\text{m} \times 9.7 \mu\text{m}$. Figure 2 is the spectrum resulting from the corresponding point EDS analysis. For this grain, a negligible carbon peak was detected. The test was repeated on a smaller grain (Fig. 3), resulting in a much larger carbon peak (Fig. 4), as expected.



Figure 1: SEM image of a relatively large Olivine grain. The electron beam was focused to a point on the sample indicated by the number “1” in the figure.

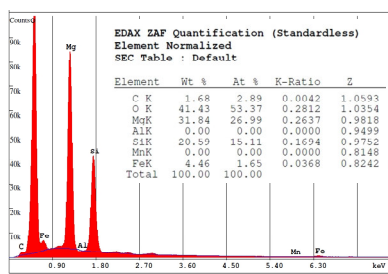


Figure 2: Spectrum of grain in Fig. 1, showing a small carbon peak.

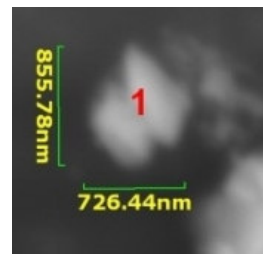


Figure 3: SEM image of a <1 μm -sized Olivine grain.

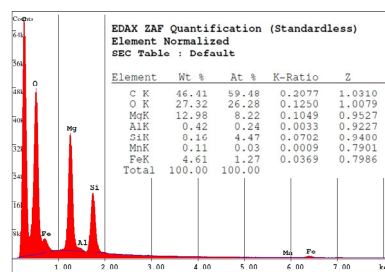


Figure 4: Spectrum grain in Fig. 2, showing a large carbon peak.

It is important to note that these analyses were performed without the use of Standards (Standardless). Therefore, the elemental values presented in Figures 2 and 4 are not absolute. We have since obtained Standard-corrected values that will be presented at the conference.

Conclusion: Future experiments will involve a more detailed analysis and quantification of mSEM EDS limitations. We will also determine our limiting X-ray detector sensitivity and correlate this to predicted measurements. Our model will be extended to include a full simulation of our secondary and backscatter electron emission. These results will be used to determine the most favorable mSEM settings (e.g., accelerating voltage and electron current at the sample) for lunar sample analysis, as well as for determining the optimum detector geometry for our mSEM assembly.

References: [1] C. M. Pieters, et al. (2009) *Science* 568 [2] http://www.nasa.gov/mission_pages/LCROSS/main/prelim_water_results.html; [3] K. G. Thaisen, et al. (2009) *40th LPSC* 1697; [4] McKay, et al. (1991) *The Lunar Sourcebook* 285; [5] Yang Liu, et al. (2007) *Am. Miner.* 1420; [6] Jaesung Park, et al. (2008) *Jour. of Aerospace Eng.* 266; [7] Sarah Nobel (2009) *Lunar Regolith Sim. 10*; [8] L. A. Taylor, et al. (2005) *36th LPSC* 1812; [9] www.FEI.com, Quanta 600 FEG Field Emission Environmental SEM specifications, from FEI; [10] Raynald Gauvin, et al. (1996) <http://www.gel.usherbrooke.ca/casino>; [11] S. Agostinelli, et al. (2003) *Nuc. Inst. and Methods in Phys. Research* 250.