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Surface elemental analysis from samples in ambient atmosphere pressure using the AEXS instrument

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This paper describes the results of the characterization experiments using the Atmospheric Electron X-ray Spectrometer (AEXS) instrument for performing elemental analysis from samples in planetary atmospheres in detail. The AEXS instrument uses a focused electron beam to excite characteristic X-Ray Fluorescence (XRF) and other luminescence spectra from samples in planetary atmospheres. The excitation in planetary atmosphere is enabled by using a thin electron-transmissive membrane to isolate the vacuum of the electron source from the outside ambient pressure. When used with a sufficiently short membrane-to-sample working distance, the transmitted electrons excite XRF spectra similar to that in SEM; however unlike in SEM, the sample does not need to be drawn into the electron source vacuum. The XRF spectra are analyzed to determine surface elemental composition for the irradiated spots with high-to-medium spatial resolution.

The feasibility of the AEXS instrument has been demonstrated in several stages, consisting of constructing and using increasingly integrated vacuum setups that simulate the AEXS operation. The first setup[1,2] (not a portable instrument) consisted of an actively pumped vacuum chamber with a 10 keV electron gun incorporated into a high vacuum feed-through at one end, and a SiN window through which the electrons were transmitted, integrated into the vacuum flange at the opposite end. In the next setup,[3] the membrane was attached directly to the exit aperture at the end of the glass tube of a commercially available 10 keV gun. The most recently assembled, "stand-alone" microprobe[4] requires no external vacuum support a big step toward the construction of a fully portable instrument. The microprobe includes an electron-optics column rated up to 20keV, a 500 nm thick SiN encapsulation membrane, has been in operation over 1 year with no observable degradation in the electron column vacuum, and has been used to acquire spectra for a number of NIST and USGS traceable metal and mineral standards in the environmental chamber that has been constructed in our laboratory to simulate the effect of reduced atmospheres on the acquired spectra. In the experiments, the spectrum analysis time was set at 100 seconds. The spectra were analyzed using a commercial SEM/EDX software package, with a good agreement obtained for the certified compositions even with no corrections for the effect of the encapsulating membrane or working atmosphere included in the analysis, up to about 90 Torr-cm atmosphere thickness. Spectral degradation that has been observed at increased atmosphere thickness can be partially remedied by including corrections to the EDX code that account for loss of energy and spectral coherence for the excitation electrons, and for X-ray absorption as the escaped X-rays travel from the sample to the detector. The results of our preliminary analysis are in agreement with the observed data, suggesting that by incorporating the membrane's and atmosphere's effects into the EDX analysis, the accuracy of the elemental determination could be improved (and possibly extended to longer distances and higher pressure). Working at reduced pressures in Martian or other NASA mission destination atmospheres will enable operation at longer working distances. To develop an instrument with low power consumption and mass for Mars exploration, we plan to replace the thermionic emitter of the present microprobe with CNT field-emitters,[5] greatly simplifying the power supply architecture.

1 References

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