



## Atmospheric Electron-Induced X-Ray Spectrometer Development@

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### 1 Introduction

This paper describes the progress in the development of the so-called Atmospheric Electron X-ray Spectrometer (AEXS) instrument in our laboratory at JPL. The AEXS is a novel miniature instrument concept<sup>[1–3]</sup> based on the excitation of characteristic X-Ray Fluorescence (XRF) and luminescence spectra using a focused electron beam, for non-destructive evaluation of surfaces of samples *in situ*, in planetary ambient atmosphere. *In situ* operation is obtained through the use of a thin electron transmissive membrane to isolate the vacuum within the AEXS electron source from the outside ambient atmosphere. By using a focused electron beam, the impinging electrons on samples in the external atmosphere excite XRF spectra from the irradiated spots with high-to-medium spatial resolution. The XRF spectra are analyzed using an energy-dispersive detector to determine surface elemental composition. The use of high-intensity electron beam results in rapid spectrum acquisition (several minutes), and consequently low energy consumption (several tens of Joules) per acquired XRF spectrum in comparison to similar portable instruments.

The use of electrons as the excitation particles enables a new approach for *in situ* XRF measurements. In the past, XRF instruments have proved to be invaluable for determining the elemental makeup of the surface of a planetary body. To date, all *in situ* missions, including Surveyor, Viking 1, Viking 2, Mars Pathfinder, Mars Exploration Rovers (MER), and the Soviet Venera/Vega missions have carried some form of XRF instrument. These instruments have used radioactive sources to provide al-

pha particles<sup>[4,5]</sup> and X-rays for excitation in planetary atmosphere. Electron beam excited energy dispersive X-ray analysis is a widely accepted technique for determining the elemental surface composition, however it has not been previously used in ambient atmosphere due to the difficulty of generating and transmitting electron beams through the atmosphere. Electron excitation for planetary exploration was considered only within the context of a miniature Scanning Electron Microscopy (SEM) that would have required preparing and placing planetary samples into the SEM's vacuum. Electron excitation using AEXS is similar to that used in SEM, with one significant advantage: Unlike in SEM, the membrane-isolation of the source vacuum obviates the need for sample to be drawn into the vacuum of the electron column. The electron probe requires no external pumping or sample manipulation. The spectra can be acquired from samples in their pristine state, since the electron-ionization of Martian atmosphere ensures a return current path so that nonconductive samples do not need to be coated with conductive material to prevent charging effects.

The applications of the AEXS instrument for NASA planetary exploration include determination of elemental abundance, or in the case of electron-induced luminescence, identification of minerals suspect of past biological activities, through correlation of x-ray and luminescence data. The penetration depth for the electrons into subsurface is on the order of several  $\mu\text{m}$ . This short penetration depth will allow surface coatings and weathering rinds on rocks to be studied with minimal mixing effects from deeper material. Measurements of unaltered surfaces will require prompt sampling of freshly cored or broken rocks, activities that are planned in several future Mars sampling missions.

## 2 Instrument Development

The proof-of-principle for the instrument is being demonstrated through 1) characterization of the effect of encapsulation membranes on the properties of the excitation beam, 2) assembly and characterization of a prototype instrument, and 3) simulation of AEXS observational capabilities. The AEXS consists of a high-energy ( $>10\text{keV}$ ) electron gun encapsulated by an electron transmissive, vacuum-isolating membrane, an EDX detection and analyzer system, an optional CL detection system, and a high voltage power supply. The effect of the membrane is determined by comparing the spectra acquired from metal and mineral samples with and without the membrane intersecting the excitation beam for samples in the laboratory ambient and in the environmental chamber. The comparison isolates the effect of membrane and atmosphere interactions, which will determine performance limits for AEXS *in situ* operation.

The prototype instrument consists of an electron gun that accelerates and focuses the electrons on the target. A breadboard stand-alone instrument requiring no vacuum apparatus support is being assembled in our laboratory. The observational capabilities are simulated through acquisition of XRF and luminescence spectra using the Scanning Electron Microscope (SEM), CL spectrometer, and an environmental chamber to simulate the effect of Mars planetary atmosphere.

**Membrane Viability.** The membrane properties were investigated in detail due to its critical role in the encapsulated instrument. A series of experiments determined that our designed membranes have high electron transmission, yet are capable of isolating high vacuum, are able to withstand differential pressure in excess of one atmosphere, survive vibrational shocks of a magnitude to be expected during a planetary mission. Typical membranes were 200 nm thick films of Silicon Nitride (SiN) microfabricated within a Si support frame. The window openings (1.5 mm x 1.5 mm) are defined by standard photolithography and reactive ion (RIE) and wet chemical etching.

The properties of the transmitted electrons are modified due to electron interaction with the molecules of the traversed medium. To determine the effect of the transmission on the excited spectra, characterization studies were performed that consisted from 1) Electron beam spreading modeling and measurements, and 2) Comparison of the excited x-ray spectra in vacuum and atmosphere without and with the intervening membrane, with emphasis on determining (i) the fraction of the transmitted electrons, (ii) beam spreading, and (iii) loss of the electron energy. Electrons suffer both elastic and inelastic collisions as they travel through a matter. Elastic collisions are essentially Rutherford scattering events and are responsible for beam divergence while inelastic collisions due to XRF generation decrease electron energy, leading to a loss of energy coherence. The results of Monte Carlo simulations<sup>[6,7]</sup> were compared with the results of beam spot size measurements conducted within an SEM with the membrane inserted into the beam path. The validated model gives us confidence to predicts AEXS spatial resolution under Martian operating conditions. For example, at a 1 cm to 10 cm working distance, the predicted spot size varies from less than 1 mm to about 1 cm, for 20kV electrons.

**AEXS prototype.** The next step in the AEXS development consisted of assembling and operating a setup that consisted of a 10 keV electron gun incorporated into a vacuum chamber (approximately  $10^{-6}$  Torr) with a SiN/Si window through which the electron beam was transmitted into the outside atmosphere. Using this setup, the robustness and vacuum sealing capability for SiN membranes has been demonstrated.<sup>[1]</sup> The setup was used to acquire XRF spectra from metal and mineral samples in an Earth atmosphere ambient that were compared with XRF spectra obtained in a laboratory SEM using both the built-in, liquid nitrogen cooled, high resolution XRF detector,

as well as the Amptek XRF silicon *p* type-intrinsic-*n* type (PIN) photodiode based detector system used on the Mars Pathfinder APXS<sup>[4,5]</sup> instrument. The SEM spectra were acquired with and without the intervening membrane in the beam path.

The XRF data was used to determine the elemental composition using the energy-dispersive X-ray (EDX) analyzer system. A good agreement was obtained between the various measurements. We were able to detect trace elements with a relative abundance of a few percent within a matrix, the X-ray transition lines were at the same locations (as they should) in all cases. However the line width was broader due to the poorer resolution of the Amptek detector, and the background was increased due to the increased electron scattering by the intervening membrane and the outside (760 Torr) atmosphere. The peaks were better resolved in the SEM due to the greater sensitivity of a liquid nitrogen cooled x-ray detector. For the Peltier cooling, the best obtainable x-ray peak width was approximately 250 eV. The irradiated spot sizes were about 1 mm and XRF acquisition times about 100 sec.

The electron gun that has been selected as our “baseline” for the development of a portable, stand-alone instrument without the support of a vacuum pump is a Thomas Electronics electron gun rated for operation at up to 20keV. The use of more energetic electrons results in reduced beam divergence (smaller spot size on the target), increased beam transmission, and the ability to excite K-shell XRF from heavier elements. The membrane thickness has been increased to 500 nm, and the aperture has been made circular, enabling a more robust vacuum-encapsulation process. The encapsulated gun is now being used to acquire spectra both in the environmental chamber and Earth atmosphere.

***AEXS Capability Simulation.*** The 10 keV setup and the 20 keV stand-alone source were used to acquire XRF spectra from metal and mineral samples in an Earth atmosphere ambient that were compared with XRF spectra obtained in a laboratory SEM using both the built-in, liquid nitrogen cooled, high resolution XRF detector as well as the Amptek XRF detector. To determine the membrane effect on the spectral count, the SEM spectra were acquired with and without the presence of an intervening membrane above the sample. The XRF yield depends on a number of factors, including the electron beam energy, beam current, chemistry and pressure of the outside atmosphere, and the relative positions and orientations of the electron source, the target and the detector. The results suggest that a 10  $\mu$ A beam will resolve the electron-excited XRF spectra in several minutes, with the irradiated spot sizes about 1 mm. The targets included standard alloys and minerals, and JSC-1 sample, and for cathodo-luminescence (CL) measurements, also samples of biogenic origin. We found good agreement between the various measurements. We were able to detect trace elements with a relative abundance of a few percent within a matrix. The line width

was broadened (as expected) due to the poorer resolution of the Amptek detector, and the background increased due to the increased scattering of the electrons by the membrane and the outside (760 Torr) atmosphere. The biogenic samples included minerals ranging in age from modern endolithic (“rock-dwelling”) microbial communities that were still living to ancient (2.1 billion year old) fossilized communities, including gypsum evaporates from Death Valley, California, and silica mineral samples from Mojave, California, National Reserve. The goal of CL measurements was to establish correlation between the elemental abundance and CL spectra, to determine regions on samples that were modified by biogenic activities. We found that regions colonized by microorganisms had often a different suite of elements, and exhibited more significant variations than the host rock, consistent with heterogeneous segregation of elements and morphologic structures in the microfossil-containing region.

### 3 Conclusions

We are developing an electron-based instrument for observation of planetary surfaces *in situ*. *In situ* observation in planetary atmospheres is enabled through using a thin electron-transmissive membrane for vacuum-isolation of the electron source.

The most significant concern associated with using the encapsulation membrane is that it degrades the spatial resolution and energy coherence of the electron beam. Although the spatial resolution of the AEXS will never be as good as the nm-sized spots of laboratory scale SEMs, it is still significantly better than any of the state-of-the-art *in situ* XRF instruments. Whereas the spatial resolution for the Alpha-Particle X-ray Spectrometer (APXS) on MER mission is on the order of several cm, the surface area irradiated by the AEXS beam can be varied from several cm to less than 1 mm by varying the “working” distance between the instrument membrane and target. In fact, the spatial dispersion for the electron beam enables two different modes, a large spot-size (cm-scale) “survey”, and a high-resolution (sub-mm scale) observation. The focusing properties of the electron beam are controlled by electron optics within the electron source. This occurs because the interaction of the transmitted electrons with the membrane and outside atmosphere leads to beam divergence (that depends on membrane composition and thickness, atmosphere pressure, and electron energy). The beam spreading can in turn be used to vary the size of the irradiated spot on the sample by simply varying the distance between the membrane and the sample. By positioning the instrument head between several millimeters to several centimeters away from the target, the spot size can be varied from sub-mm to several cm on the Mars atmosphere.<sup>[6]</sup> Using a relatively large working distance (10 cm-scale on Mars) the

size of the irradiated spot can be cm-scale, making it possible to use the AEXS instrument for surface reconnaissance. When an area appears interesting, the instrument head could be brought close to the surface for a more detailed inspection and surface maps acquired during instrument head stepping. Rapid spectrum acquisition is needed to use the high-resolution capability for the electron beam. Rapid acquisition is enabled due to the combination of a large flux and high excitation efficiency using an electron beam. Electron-induced excitation is a one-step process, leading to spectra acquisition times less than 1 minute for a  $10\mu\text{A}$  beam (as compared to about 1 hour for APXS). Such short times will also result in low energy consumption per spectrum and thus enable multiple readings assessing sample heterogeneity.

We are also planning to replace the thermionic emitter with a Carbon-nanotube-based field-emitter,<sup>[8]</sup> thus greatly simplifying the power supply architecture, leading to additional savings in mass as well in a significantly lower power consumption. The predicted mass and size for the completed instrument are on the order of 1 kg and 10cm-scale scale dimensions, respectively.

## 4 References

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