

ATMOSPHERIC ELECTRON-INDUCED X-RAY SPECTROMETER (AEXS) INSTRUMENT DEVELOPMENT. J.Z. Wilcox¹, E. Urgiles¹, R. Toda¹, and J. Crisp¹, ¹Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, 302-231, Pasadena, CA 91109 (Jaroslava.Z.Wilcox@jpl.nasa.gov).

Introduction: The progress in the development of the AEXS is described. The AEXS is a surface analysis tool based on excitation of characteristic x-ray fluorescence (XRF) spectra from samples in ambient atmospheres using a focused electron beam.^{1,2} Operation in ambient atmospheres with moderate-to-high spatial resolution in comparison to similar instruments is obtained through the use of a thin electron transmissive membrane to isolate the vacuum of the electron probe, obviating the need for the samples to be drawn into the probe vacuum. The AEXS system (Fig 1) consists of an electron microprobe encapsulated by the isolation membrane, and an EDX detection and analyzer system to determine the elemental abundance. Our initial, previously reported setup³ that was used for the demonstration of the ability of the transmitted electrons to excite XRF spectra in the external atmosphere, consisted of an actively pumped chamber from within which the electrons were transmitted - not a portable instrument. The AEXS instrument that has been assembled in our laboratory during the past year consists of a 20keV electron gun vacuum-sealed with a thin SiN membrane, and requires no active pumping - a big step towards the development of a stand-alone instrument. The microprobe has been in operation for over 1 year and used for performing rapid elemental analysis of NIST and USGS traceable metal and mineral standards, with good agreement with the certified composition for samples in up to about 90 Torr-cm thick external atmospheres.

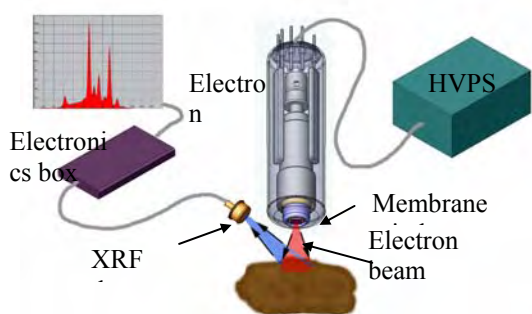


Fig. 1. The AEXS instrument concept. The AEXS consists of a miniature, vacuum-encapsulated electron source, HVPS, and XRF detector. The electron beam is transmitted through the electron-transmissive vacuum-isolation membrane into the ambient atmosphere, strikes the sample, exciting characteristic XRF spectra for elemental analysis of the irradiated spot. The spot area can be adjusted from sub-mm to several cm size by changing the membrane-sample working distance.

The approach towards instrument development has been through 1) demonstrating the viability of micro-fabricated membranes, 2) assembling AEXS setups with increasingly integrated functional components, and 3) simulating the AEXS observational capabilities. This paper focuses on instrument assembly.

Instrument Development: The AEXS has been demonstrated in several stages, consisting of increasingly integrated vacuum setups that simulate the AEXS operation, and show continued progress towards the construction of a portable instrument. The first setup constructed for demonstration of the ability of the transmitted electrons to excite XRF spectra, 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. The membrane bridged a $(1.5 \times 1.5) \text{ mm}^2$ opening created within a Si substrate using standard photolithographic techniques. In the next setup, the membrane was attached directly to the exit aperture at the end of the glass tube of the 10 keV gun. The gun, operated under the support of a high vacuum pump via an exhaust port, was used to excite XRF spectra from metal and mineral samples that were compared with spectra acquired within the SEM, with an excellent agreement obtained for the positions of the spectral peaks.^[3] The most recent microprobe differs from the previous setups in that it requires no active pumping. The microprobe (Fig 2) envelops an electron-optics column within a glass tube that has been vacuum-sealed using a specially designed glass-tube attachment that incorporates the transmissive membrane. Because of the lack of need for active pumping, the microprobe provides for development of a fully portable instrument.

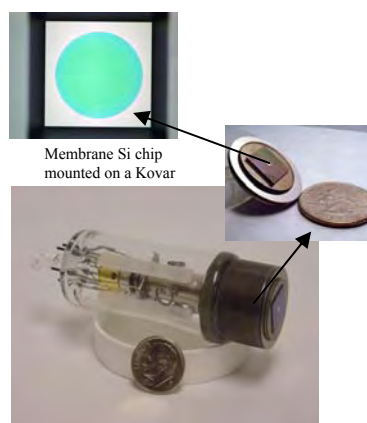


Figure 2. Vacuum-Isolated 20keV Electron source

The microprobe was used to excite XRF spectra from a number of known NIST and USGS metal and mineral standards within an environmental chamber (Fig 3) that has been constructed in our laboratory to



Figure 3. Environmental Chamber integrated with the Electron gun and Amptek

simulate the effect of reduced atmospheres on the acquired spectra. Using a 10 μ A beam, the spectra were resolved with the analysis time set at 100 seconds (the same as our standard procedure in our SEM lab). The microprobe has been in operation over a year with no observable

degradation in the gun vacuum Fig 4 shows the acquired spectra for a BCR-2 (Basalt, Columbia River) mineral sample for a set of Nitrogen pressures varying from 10^{-4} Torr to 200 Torr.

Discussion. The spectra were analyzed using a commercially available EDX Quantification software acquired from IXRF corporation, and used by many laboratory SEM systems to analyze the XRF spectra. Upon the specification of the beam energy and take-off angle, and specifying the elements list for the analysis, the software calculates the required line energies and

the elemental abundance by using Gaussian deconvolution for peak extraction and ZAF corrections for the quantitative analysis.

In our experiments, the abundances were determined by performing "standard-less" quantification. That is, no side-by-side standards were provided for additional calibration measurements. The results of the measurements using samples with known composition (i.e. traceable standards) establish limits on the instrument's performance. Figure 4b shows the spectra after the subtraction of the Bremsstrahlung background. The computed elemental abundance for the BCR-2 sample and accuracy of the analysis are discussed in the adjoining paper.⁵ The agreement between the determined and certified abundances was good, especially considering the uncertainties in the analysis, up to about 90 Torr Nitrogen pressure. At higher pressure and for the 1 cm working distance, the discrepancy between the certified and experimental abundance becomes greater than 50% for most of the analyzed elements.

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. The effect of the membrane and the intervening atmosphere has been analyzed with good agreement with the observations. We are also planning to replace the thermionic emitter with a Carbon-nanotube-based field-emitter, thus greatly simplifying the power supply architecture, leading to additional savings in mass as well in 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.

References: [1] J. Feldman, J.Z. Wilcox, T. George, D. Barsic, and A. Scherer, "Elemental Surface Analysis at Ambient Pressure by Electron-Induced X-ray Fluorescence", *Rev. Sci. Instr.*, March 2003; [2] J.Z. Wilcox, E. Urgiles, T. George, J. Crisp, E. Baumgartner, "Atmospheric Electron Induced X-ray Spectrometer (ELXS) for exploration of samples on Martian surface *in-situ*", NASA's '04 MIDP program; [3] E. Urgiles, J.Z. Wilcox, R. Toda, J. Crisp, and T. George, "Characterization of the XRF performance of the Atmospheric Electron Induced X-ray Spectrometer (AEXS)", presented at LPSC'05; [4] E. Urgiles, R. Toda, J.Z. Wilcox, "AEXS Development", *Rev. Sci. Instr.*, Jan 2006; [5] E. Urgiles, J.Z. Wilcox, R. Toda, and J. Crisp, "Elemental composition analyses in thick atmosphere using the Atmospheric Electron X-ray Spectrometer (AEXS)", LPSC'05.

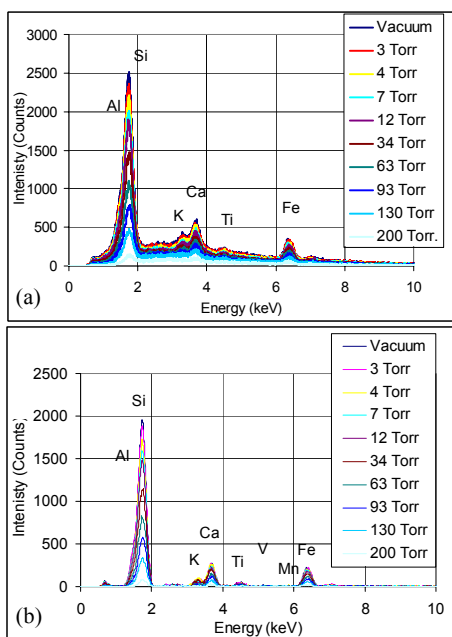


Figure 5. XRF spectra for the BCR-2 (Basalt, Columbia River) mineral sample taken using the vacuum-isolated 20 keV electron gun in the environmental chamber at several values of the ambient pressure: (a) with background, (b) subtracted background. (The spectra are shown in the order of increased pressure, with the vacuum at top).

resolutions for the specified analyte lines, subtracts the best-fit Bremsstrahlung background, and determines