

ATMOSPHERIC ELECTRON-INDUCED X-RAY SPECTROMETER (AEXS) INSTRUMENT DEVELOPMENT. E. Urgiles¹, J.Z. Wilcox¹, R. Toda¹, J. Crisp¹, and T. George¹, ¹Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, 302-231, Pasadena, CA 91109 (Eduardo.Urgiles@jpl.nasa.gov).

Introduction: This paper describes the progress in data acquisition and establishing the observational capability of the AEXS instrument. The AEXS is a miniature instrument^[1-4] based on the excitation of characteristic X-Ray Fluorescence (XRF) and luminescence spectra using a focused electron beam which enables non-destructive evaluation of sample surfaces in planetary ambient atmospheres. *In situ* operation is obtained through the use of a thin electron transmissive membrane to isolate the vacuum of the AEXS source from the outside ambient atmosphere. Thus eliminating the need for a vacuum pumped sample chamber as is common in all laboratory SEM's. The transmitted electrons impinge on the sample exciting XRF spectra from the irradiated spot on in-situ or collected samples with sub-mm to cm-scale spatial resolution at Mars atmospheric pressure. The AEXS system (Fig 1) consists of a high-energy (>10keV) electron gun encapsulated by the isolation membrane, an XRF detection and analyzer system, and a high voltage power supply. The XRF data are analyzed to determine the elemental abundance for the irradiated spots. The approach to demonstrating a proof of concept of the AEXS 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. The development of the instrument is described in detail in the poster paper^[4] at this conference. This paper focuses on describing the progress in establishing the capability of the AEXS instrument to acquire XRF

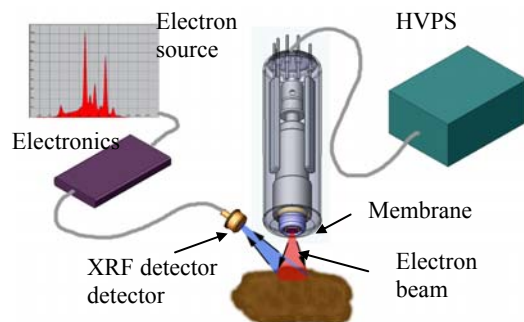


Fig. 1. The AEXS instrument concept. The AEXS consists of a miniature, vacuum-encapsulated electron source, HVPS, and XRF detector. The electron beam is transmitted through the electron-transmissive vacuum-isolation membrane into the ambient atmosphere, strikes the sample, exciting characteristic XRF 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.

data and using commercially available software to analyze the data streams and determine the accuracy, precision and resolution of the analysis compared to the certified elemental abundance.

Data Acquisition: Our initial work, with a 10 keV electron gun that used a 200 nm thick Silicon Nitride (SiN) vacuum-isolation membrane microfabricated within a Si support frame with (1.5 mm x 1.5 mm window openings), confirmed the ability for the electron beam transmitted through the membrane to efficiently excite XRF spectra and determine elemental abundance for the irradiated spots using energy-dispersive analysis of the excited spectra. Figure 2 shows spectra taken with the 10 keV gun and compares them with spectra taken within SEM for Waspaloy, a NIST traceable high temperature alloy. All peaks occur at their

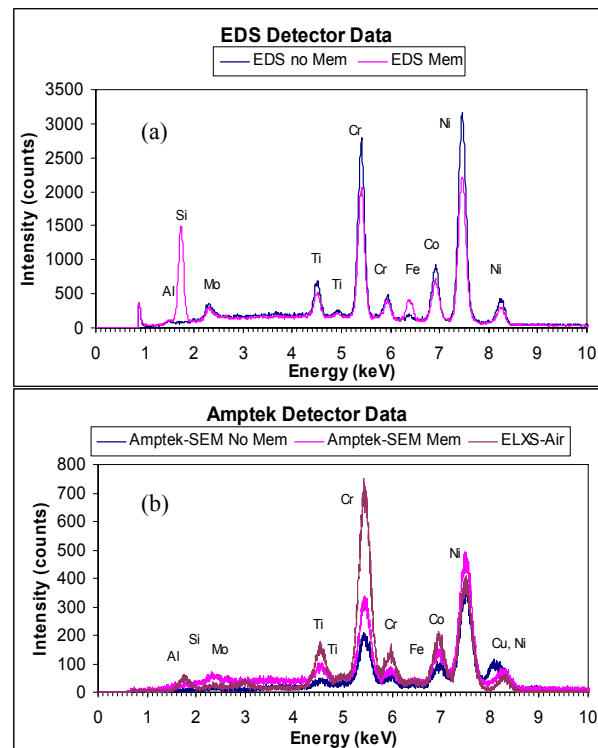


Fig. 2. X-ray spectra obtained from a Waspaloy (about 59%Ni, 19% Cr, 12% Co, 4% Mo, 3%Ti) standard. (a) in SEM without and with the intervening membrane using EDS detector, (b) in SEM and the AEXS setup (Fig 5) using the Amptek detector and 10 kV electron gun. All peaks occur at their correct locations, Si peaks occur due to electron-membrane window interaction, and lines in (b) are broadened due to the comparatively poor resolution of the detector.

correct locations, although they are broadened due to the poor resolution of the Amptek (vs liquid Nitrogen cooled SiLi, SEM EDX) detector.

Following the 10 keV gun experiments, we have concentrated on the development of a portable, stand-alone instrument (without the support of a vacuum pump), using a Thomas Electronics electron gun rated for operation up to 20keV. The use of more energetic electrons results in reduced beam divergence (smaller spot size on the target), increased beam transmission, the ability to excite K-shell XRF from heavier elements, and increased robustness due to the possibility to increase the membrane thickness to 500 nm. The encapsulated gun has been used to acquire spectra both in the environmental chamber and Earth atmosphere.^[5] For our current study we shifted our sample selection from the NIST traceable metal alloys which we used in our 10keV experiments to USGS traceable powders. This serves the purpose of establishing the analytical performance of the instrument with non-conductive soil samples more closely resembling the rocks and soils to be found on planetary surfaces. We have challenged the instruments with the task of identifying different Basalts (Columbia River Basalt BCR-2 & Icelandic Basalt BIR-1) as well as characterizing marine sediments (MAG-1), a sulfur & organic rich shale (SDO-1), and ultra mafic alkaline and carbonate rocks (COQ-1).

Discussion. One of the concerns associated with using an electron excitation beam that traverses the encapsulation membrane and outside atmosphere between the membrane and target is the accuracy and sensitivity with which the XRF spectra can be analyzed to determine the elemental abundance. The results of our measurements show that although the membrane and atmospheric constituents do contribute to the overall x-ray signals collected by the detector. It is relatively easy to mathematically correct for the difference in composition due to the atmosphere's contribution and the transmission through the membrane.

Another concern associated with using the membrane is that it degrades the spatial resolution and energy coherence of the electron beam due to the interaction with the transmitted electrons. 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

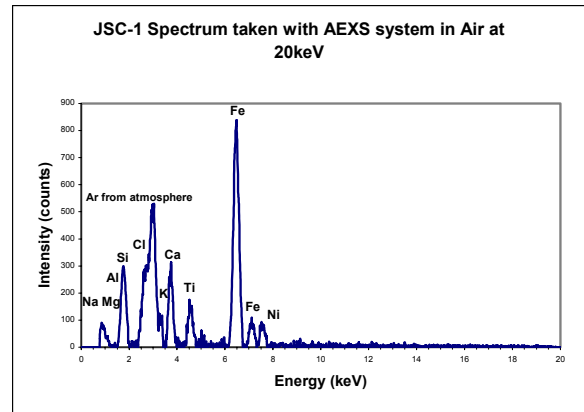


Fig 3 shows the spectra from a sample pellet of JSC-1 composite taken with the 20 keV gun at atmospheric pressure.

cm to less than 1 mm by varying the “working” distance between the instrument membrane and target. In fact, the spatial dispersion for the 20 keV beam in our experiments has resulted in sub-mm spot sizes on targets at a 1 cm working distance from the membrane in Martian atmosphere pressure. Using 10 μ A beams, the spectra acquisition times were less than 1 minute (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.

References: [1] J.E. 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.E. Feldman, S. Douglas, “*Electron Induced Luminescence and X-ray Spectrometer (ELXS) Development: Progress Report*”, presented at the Aerospace IEEE’03 conference, and reprinted in the conference proceedings; [3] J.Z. Wilcox, E. Urgiles, S. Douglas, and T. George, “*Electron Induced Luminescence and X-ray Spectrometer (ELXS) Development*”, presented at the LPSC’04 conference; [4] 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; [5] J.Z. Wilcox, E. Urgiles, R. Toda, T. George, and J. Crisp, “*Development of the Atmospheric Electron Induced X-ray Spectrometer (AEXS)*”, a poster paper at this conference;