

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

Introduction: The AEXS is a miniature instrument^[1-4] based on the excitation of characteristic X-Ray Fluorescence (XRF) spectra from samples in ambient atmosphere using a focused electron beam. Operation in ambient atmosphere is obtained through the use of a thin electron transmissive membrane to isolate the vacuum of the AEXS electron probe from the outside atmosphere. The isolation obviates the need for the samples to be drawn into a vacuum pumped sample chamber as is common in all laboratory SEM's. The microprobe requires no active pumping – enabling to construct a portable instrument. The transmitted electrons through the membrane impinge on samples in the external atmosphere, exciting XRF spectra from samples in their pristine state with no surface preparation, with sub-mm to cm-scale spatial resolution at Mars atmospheric pressure. The spectra are analyzed to determine elemental abundance for the irradiated spots. The development of the encapsulation procedure has been described in details previously.^[1-4] We focus on describing the capability of the AEXS to resolve surface elemental composition with mm-scale spatial resolution using a commercially available software to analyze XRF spectra. The progress in the development of an instrument head, consisting from an integrated high voltage power supply with the electron microprobe is discussed.

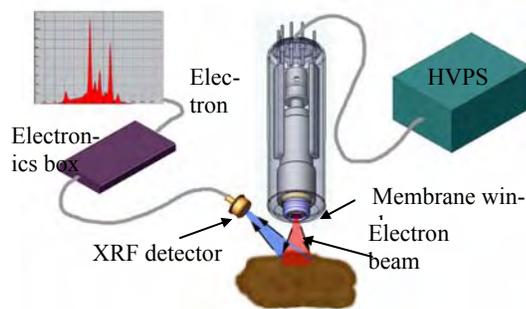


Fig. 1. The AEXS consists of a miniature, vacuum-encapsulated electron source, HVPS, and XRF detector. The electron beam is transmitted through the electron-transmissive vacuum-isolation membrane into the ambient atmosphere, strikes the sample, exciting characteristic XRF spectra for elemental analysis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

Electron Microprobe: The AEXS system (Fig 1) consists of an electron microprobe vacuum-sealed with

a thin SiN encapsulation membrane, an EDX detection and analyzer system, and a high-voltage power supply and associated electronics. The microprobe (Fig 2) consist of an electron-optics column enveloped within a glass tube that has been vacuum-sealed using a specially designed attachment that incorporates the transmissive membrane. The emitted electrons are focused and accelerated to 20 keV inside the electro-optics column. The membrane is a 500 nm thick SiN film microfabricated using standard photolithographic techniques to bridge a (1.5×1.5) mm² opening within a Si substrate.

Spectra acquisition. The microprobe was used to

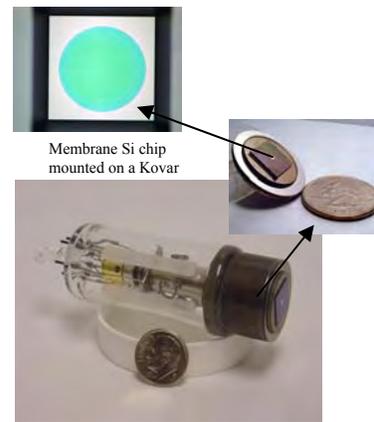


Figure 2. Vacuum-Isolated 20keV Electron source

excite XRF spectra from a number of known NIST and USGS metal and mineral standards within an environmental chamber that has been constructed to simulate the effect of reduced atmospheres on the acquired spectra. The spectra were analyzed by using a commercially available

EDX Quantification software acquired from IXRF corporation. As a way of example, Fig 3 shows the spectra for a BCR-2 (Basalt, Columbia River) mineral sample for a set of Nitrogen pressures varying from 10⁻⁴ Torr to 200 Torr. The spectra were resolved with the analysis time set at 100 seconds (the same as our standard procedure in our SEM lab) using several μ A beam current. The membrane-to-sample working distance was 1 cm. The derived abundances were compared with the provider-certified composition. The agreement was good up to about 90 Torr Nitrogen pressure. At higher pressure the values differed, due to the increased effect of the atmosphere on both the excitation electrons and excited X-rays. The spectral degradation at high 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.

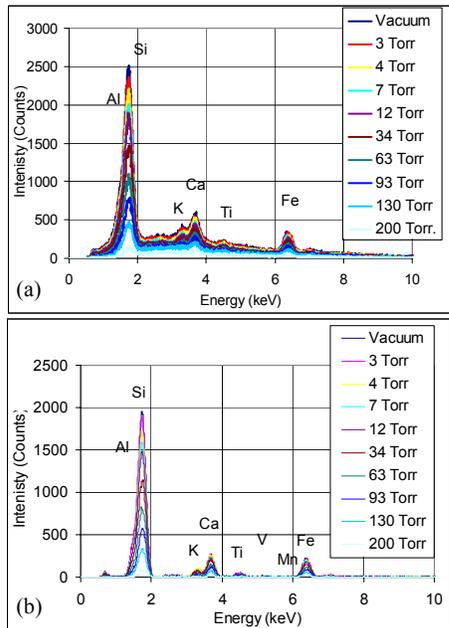


Figure 5. XRF spectra for the BCR-2 (Basalt, Columbia River) mineral sample taken using the vacuum-isolated 20 keV electron gun in the environmental chamber at several values of the ambient pressure: (a) with background, (b) subtracted background. (The spectra are shown in the order of increased pressure,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

Spatial Resolution: The spatial resolution for the microprobe was tested by performing measurements in another environmental chamber at a 1 mm working distance. Figure 4 shows a Gabbro (Norite) sample from the Stillwater Complex, Sweet Grass County Montana, which contained mineral grains of 1 to 3 mm in size. The AEXS beam differentiated the grains with about 1 mm spatial resolution. Specifically, as Figure 4 shows, Mg & Fe rich areas were differentiated from Ca & Al rich areas in the rock. This is the first demonstration of the ability of any XRF instrument to map surface elemental composition with better than several cm-scale spatial resolution. Other XRF instruments (APXS, flown on Pathfinder and MER) or the CheMin (selected for MSL mission), are essentially bulk analysis instruments. Resolving the elemental composition spatially may give clues about sample's geologic origin.

Instrument head for a mobile platform. The AEXS development includes assembly of an instrument head that integrates a Walton-Cockroft high voltage multiplier to deliver voltages to the gun's four electrodes, and to the filament heating power supply. When implemented on a mobile platform, AEXS would be able to determine elemental composition of freshly exposed rock surfaces or soil grains on planetary surfaces, as a part of a payload that would also include a

visual light camera capable of imaging the area being analyzed. The power supply is being developed in several stages, including development of electronic boards, dielectric housing (necessitated by low electrical strength of the Mars atmosphere pressure), and design for low temperature operation.

CONCLUSIONS

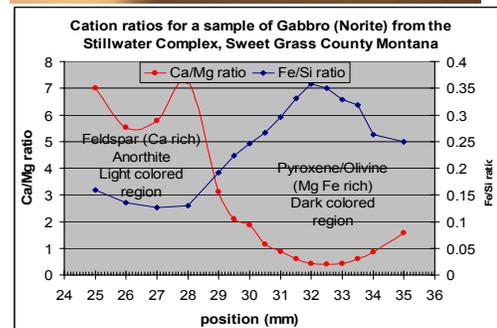


Fig 4. The AEXS instrument was used to resolve composition of grains in Gabbro (Norite) sample from the Stillwater Complex, Sweet Grass County Montana, with 1.5 mm spatial resolution. (a) The Gabbro sample contains grains of 1-3 mm in size. (b) The AEXS differentiated the Mg & Fe rich areas from the Ca & Al rich areas. Resolving the elemental composition spatially may give clues about sample's geologic origin.

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'06; [4] E. Urgiles, R. Toda, J.Z. Wilcox, "AEXS Development", *Rev. Sci. Instr.*, Jan 2006; [5] J.Z. Wilcox, E. Urgiles, R. Toda, and J. Crisp, "Elemental composition analyses in thick atmosphere using the Atmospheric Electron X-ray Spectrometer (AEXS)", LPSC'06.