

**PROGRESS IN THE DEVELOPMENT OF THE ATMOSPHERIC ELECTRON-INDUCED X-RAY SPECTROMETER (AEXS) INSTRUMENT WITH HIGH SPATIAL RESOLUTION FOR SURFACE ELEMENTAL ANALYSIS OF SAMPLES IN PLANETARY ATMOSPHERE,** E. Urgiles<sup>1</sup>, J.Z. Wilcox<sup>1</sup>, R. Toda<sup>1</sup>, J. Crisp<sup>1</sup>, <sup>1</sup>Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, 302-231, Pasadena, CA 91109 (Eduardo.Urgiles@jpl.nasa.gov).

**Introduction:** One of the most powerful techniques for the characterization of mineral samples is Electron-induced Energy-Dispersive X-ray Fluorescence Spectroscopy (EDX-XRF). If used for resolving the elemental composition for samples in their natural state, high spatial resolution data will provide insight into geological processes and formation mechanisms of planets and other solar system objects. By correlating XRF with other spectra (eg XUV and optical luminescence) additional information could be obtained about the bonding structure and oxidation states for the minerals. To date, all *in situ* missions have carried some form of an XRF instrument. For example, the APXS instrument aboard the Mars Pathfinder and MER determined bulk” averaged elemental composition over areas of several cm in diameter. The spectrum acquisition time required to resolve the XRF spectrum was several hours. In order to obtain spatial maps, the spectrum acquisition time must be decreased and the spatial resolution increased for future instruments in comparison to the current methods.

**AEXS principles:** The AEXS is a miniature microprobe<sup>[1-4]</sup> for excitation of characteristic XRF spectra from samples in planetary atmospheres rapidly (< 1min per resolved XRF spectrum) with variable and high spatial resolution (sub-millimeter to cm level) using a focused beam of energetic electrons (>20kV). For samples on Mars surface, the electron source vacuum must be isolated from the outside atmosphere. For the AEXS instrument, this is obtained by using a thin, electron-transmissive micro-machined membrane to bridge the aperture of the electron source. Though similar in the excitation principles, unlike in the Scanning Electron Microscope (SEM) technique the samples are not drawn into the electron source vacuum. The samples remain in Mars atmosphere. 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. For samples in vacuous environments (such as Moon or Mars satellite Phobos) no isolation membrane is required, alleviating beam spreading (which is caused by interaction of the excitation electrons with the membrane and outside atmosphere), further increasing the spatial resolution to the electron optics level.

**AEXS electron microprobe:** The AEXS microprobe (Fig 1) consists of an electron tube vacuum-sealed with a thin (500nm thickness) SiN encapsulation membrane, an EDX detection and analyzer system, and a high-voltage power supply and associated electronics. The emitted electrons from the microtube

are focused and accelerated to 20 keV inside the electro-optics column. The AEXS breadboard has been demonstrated in several stages, including simulation of operation, characterization of the effect of electron-transmissive

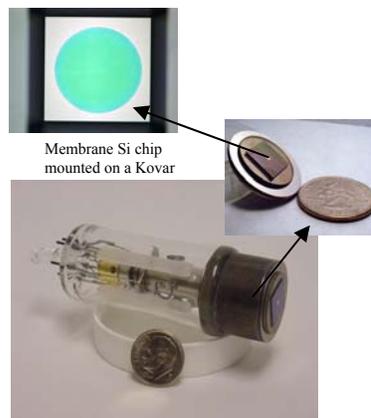


Figure 1. Vacuum-Isolated 20keV Electron source

membranes on the collected XRF spectra, assembly, and characterization of science capabilities. The developed, vacuum-isolated microprobe is “portable” in the sense that it requires no active vacuum pumping.

**Spectra acquisition.** The microprobe was used to 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 to determine the elemental abundance for the irradiated spot. The accuracy of the derived abundance was discussed previously. The spectra were resolved with the analysis time set at 100 seconds (the same as our standard procedure in our SEM lab) using several  $\mu\text{A}$  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.

**Spatial Resolution:** The spatial resolution for the microprobe was tested by performing measurements at a 1 mm working distance. As a way of example, Figure 2 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, 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.

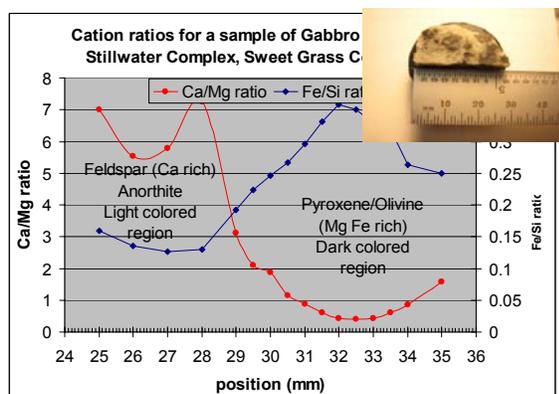


Figure 2. The AEXS instrument was used to resolve composition of Mg & Fe rich areas from the Ca & Al rich areas grains in Gabbro (Norite) sample from the Stillwater Complex, Sweet Grass County Montana, with 1.5 mm spatial resolution. Resolving the elemental composition spatially may give clues about sample's geologic origin.

**Instrument head for a mobile platform.** The selection and operation of certain instruments on future NASA missions depends on the availability of miniature High Voltage Power Supplies (HVPS) to power the instruments. These include CheMin on MSL and Urey for ExoMars, as well as the AEXS instrument. NASA missions are characterized by extreme environments that put constraints on observational methods, and in the case of landed missions, on size, mass and power consumption. The difficulties posed by extreme environments are unique to NASA, not addressed by the commercial industry. Figure 3 shows a concept for the integrated AEXS instrument head for accommodation onto the instrument arm. The instrument head consists of a miniature HVPS integrated with the electron microprobe within a housing envelope, which isolates all high-voltage (HV) components from the outside atmosphere. The HVPS includes electronic circuit boards for High-Voltage Multiplier (HVM) and control electronics. To date, we have assembled a miniature HVPS rated for operation in Earth environment (room temperature, one atmosphere, tested submerged in oil for dielectric insulation, not a flight-qualifiable instrument). To produce a flight-

qualifiable design, the objective of an ongoing investigation is to design, fabricate, encapsulate and test HV properties of test coupons (circuit boards) over an extreme temperature range, for a range of candidate encapsulation approaches, with emphasis on solid potting of surface mount components on HVM boards.

**Conclusions.** 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, yielding information at scale length of mineral grains. To decrease power consumption and mass, the thermionic emitters in the present microprobe should be replaced with Carbon-nanotube-based field-emitters,<sup>[5]</sup> greatly simplifying electrostatic focusing and power supply architecture.

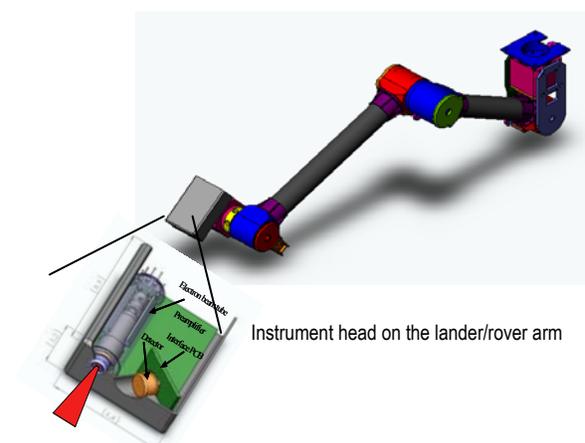


Figure 3. The size of the irradiated spot on target varies from 100  $\mu\text{m}$  to several cm depending on working distance and atmosphere

**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'07; [4] E. Urgiles, R. Toda, J.Z. Wilcox, "AEXS Development", *Rev. Sci. Instr.*, Jan 2006; [5] H.M. Manohara\*, M.J. Bronikowski, E. Wong, R. Toda, R.H. Lin, E. Schlecht, E.M. Luong, J.W. Wilcox, "CNT-based Vacuum And Semiconducting Devices For Micro-Instrumentation And Electronics," Fourth International Planetary Probe Workshop, Pasadena, CA, June 2006.