

AMBIENT-PRESSURE X-RAY PHOTOEMISSION SPECTROMETER FOR CHEMICAL ANALYSIS OF PLANETARY SURFACES. P. J. Grunthaner¹, F. Grunthaner¹, V. White¹, C. Bryson², W. Wu², M. Kelly³, and R. Quinn⁴, ¹Jet Propulsion Laboratory, MS 302-205, Pasadena, CA 91109 (paula.grunthaner@jpl.nasa.gov), ²Apparati, Inc. 221 Carpenter Dr., Hollister, CA 95023, ³Stanford University, ⁴SETI Institute, NASA Ames Research Center, MS 239-4, Moffet Field, CA 94035

Introduction: The exposed outcrops and chemical sediments of Mars retain clues to its past history—the rock weathering processes, the presence and perseverance of liquid water, and possible biological habitability. We present a design and initial test results for a low mass, low-power ambient-pressure X-ray photoemission spectrometer (XPS) for the quantitative analysis of the composition and chemical state of elements on planetary surfaces. This instrument targets high priority science objectives for Mars such as understanding geochemical processes especially with respect to the role of water, past habitability, oxidants, organic degradation, and dynamic atmosphere/mineral interactions. It is also suitable for other environments including Europa and small bodies.

XPS provides both quantitative elemental and chemical state information through the energy analysis of photoelectrons emitted from a sample that is irradiated with soft X-rays. The key strengths of XPS lie in the return of detailed chemical bonding information, quantitative chemical analysis, and sensitivity to the first 5 to 10 nm of the surface of the sample. XPS can provide direct chemical information on all elements, except H, in rocks and soils. In addition to quantifying the distribution oxidation states in a sample, XPS provides direct information on the chemical bonding, allowing the identification of chemical compounds. It is well suited for identifying and distinguishing the various possible chemical states of elements of importance to understanding the Martian outcrops and chemical sediments such as O, N, P, Mg, Ca, Cr, S, Fe, Cl, Br, and C. XPS has not previously been considered for planetary exploration because laboratory XPS systems are large (>1000 kg) power consuming instruments.

The particular implementation of XPS discussed here has been dubbed “ChemStat”, a contraction in reference to the chemical state information this technique will provide.

Key Technologies and Science Enabled: Two new technology advancements enable the miniaturization of XPS for planetary applications: i) a new electron energy analyzer and ii) a dual-thickness X-ray and electron transmissive membrane. Collectively, these two advancements enable a new XPS system that is ~100 times less massive and power consuming, without sacrificing spectral or spatial resolution. The sample remains outside the spectrometer vacuum chamber, eliminating the need for prepare and transport the sam-

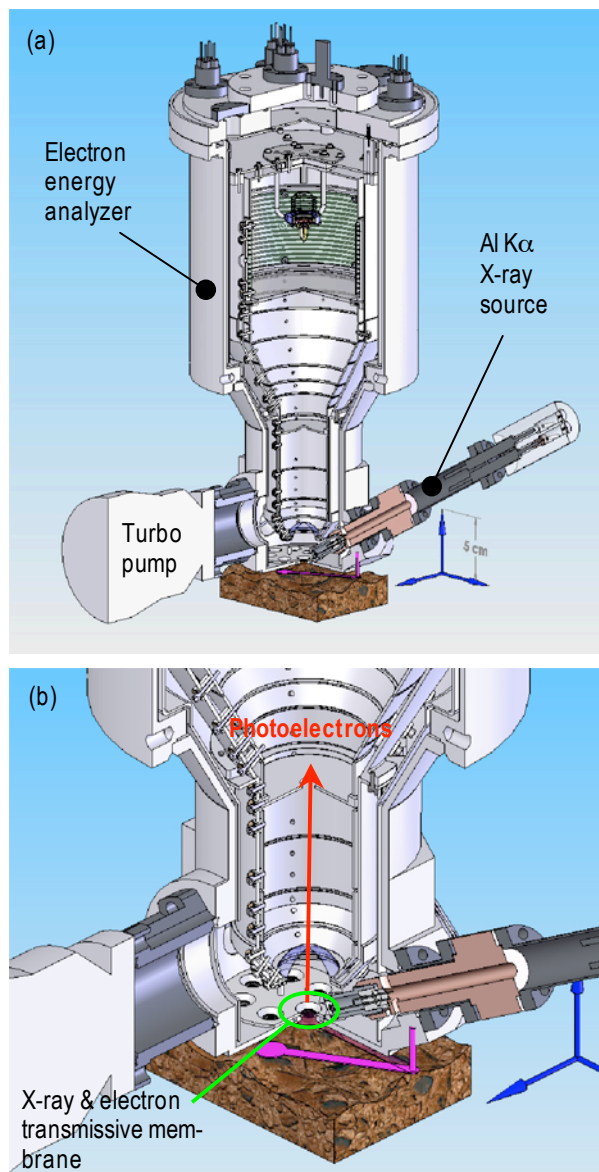


Figure 1: Ambient-pressure X-ray photoemission can provide elemental analysis as well as chemical state information for all elements except H without sample handling requirements. It probes the near-surface region (<10 nm) of a sample, including the atmospheric species interacting with the surface.

ple into the spectrometer. Figure 1 shows a schematic of the analyzer design and spectrometer concept.

Keeping the sample in its ambient environment enables one to probe in real time dynamic chemical

changes on the sample surface as a result of atmosphere/solid surface interactions. This promises new insight into such phenomenon as weathering processes, oxidant formation, photolytic or chemical degradation of organic compounds, including, for example, possible biomarkers and polycyclic aromatic hydrocarbons from meteoritic infall.

Electron Energy Analyzer. Commercial XPS systems use dispersive electron energy analyzers, which are not amenable to miniaturization because sensitivity and energy resolution depend upon the size of the concentric hemispheres typically used. In contrast, the

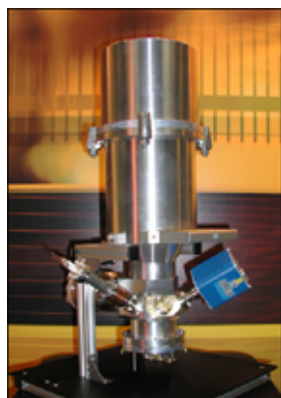


Figure 2: Electron energy analyzer breadboard.

new electron energy analyzer discussed here does not spatially disperse electrons according to their energy, but instead uses high-pass and low-pass filters to illuminate the detector with a narrow range of electron energies. This approach is amenable to miniaturization without sacrificing performance and was developed to the breadboard stage by Apparati, Inc. via a NASA SBIR. [1]

Dual-thickness Membrane. Only recently has ambient-pressure XPS emerged as a laboratory instrument and such instruments depend upon extensive differential pumping. The ChemStat implementation uses a dual-thickness silicon nitride membrane as a gas isolator film. A dual-thickness membrane is required because both X-rays and electrons must be transmitted. X-rays can easily penetrate many microns of Si_3N_4 , but electrons are unable to transmit through films thicker than 5–6 nm. Such ultra-thin films cannot be made over the required mm-size areas.

Our solution was to develop a dual-thickness that consists of arrays of ultra-thin (2.5 to 5 nm) Si_3N_4 windows on a supporting thin (1 μm) Si_3N_4 membrane, which, in turn, is fabricated on a supporting thick (300 μm) Si frame. [2] Figure 3 shows such a dual-thickness membrane (frame not in image). The rounded dome of the ultra-thin membrane provides mechanical strength and we have successfully tested dual-thickness membranes with 3.5-nm ultra-thin windows fabricated over a 25-mm² area against 35-psi pressure differential while demonstrating theoretically predicted electron transmission properties.

Spectrometer System Characteristics. Current performance characteristics estimated for a flight version of ChemStat are as follows: 7 kg, 10 W, 0.8 eV spec-

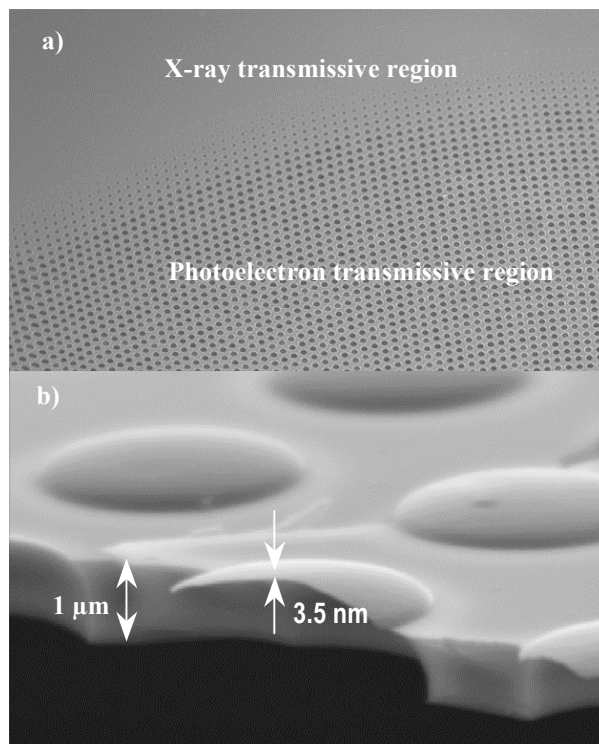


Figure 3: The dual-thickness membrane consists of a thin 1- μm -thick Si_3N_4 layer transparent to X-rays and an area with windows of ultra-thin ~3-nm-thick Si_3N_4 that allows transmission of the photoelectrons. The 300- μm -thick Si frame is not shown.

tral resolution, 100 μm spatial resolution, and operating pressure for sample of $<10^{-6}$ to 10 torr.

Acknowledgements: Part of this work was carried out at the Jet Propulsion Laboratory, California Institute of Technology, under JPL discretionary funding and later under a contract with NASA; and at Apparati, Inc., under a NASA Small Business Innovative Research Contract.

References:

- [1] Bryson C., Kelly M., Wu W. (2006), SBIR report for Contract NNC04ACA20C, 28 Feb 2006
- [2] Bryson C., Grunthaner, F., Grunthaner P., (2004) US Patent No. 6,803,570 B1