Physical Sciences

## Micro Electron MicroProbe and Sample Analyzer

## EDX and high-resolution microscopy could be performed in the field.

NASA's Jet Propulsion Laboratory, Pasaden a, California

A proposed, low-power, backpack-sized instrument, denoted the micro electron microprobe and sample analyzer (MEMSA), would serve as a means of rapidly performing high-resolution microscopy and energy-dispersive x-ray spectroscopy (EDX) of soil, dust, and rock particles in the field. The MEMSA would be similar to an environmental scanning electron microscope (ESEM) but would be much smaller and designed specifically for field use in studying effects of geological alteration at the micrometer scale. Like an ESEM, the MEMSA could be used to examine uncoated, electrically nonconductive specimens. In addition to the difference in size, other significant differences between the MEMSA and an ESEM lie in the mode of scanning and the nature of the electron source.

The MEMSA (see figure) would include an electron source that would focus a beam of electrons onto a small spot on a specimen, which would be mounted on a three-axis translation stage in a partly evacuated sample-exchange chamber. Whereas the electron sources in other SEM s typically contain thermionic cathodes, the electron source in the MEMSA would contain a field-emission cathode containing a planar array of bundles of carbon nan-
otubes (CNTs). Cathodes of this type are capable of high current densities (tens of amperes per square centimeter) at very low fields ( 8 to $10 \mathrm{~V} / \mu \mathrm{m}$ ), and the arrays of bundles of CNTs in them are amenable to fabrication within designated areas of the order of a few square nanometers, making it possible to focus electron beams to small spots using simplified electronbeam optics. Another advantage of CNT field-emission cathodes is that they can tolerate operation in relatively poor vacuums [pressures of $10^{-5}$ to $10^{-4}$ torr (about 0.013 to 0.13 Pa )], which can be maintained by relatively small turbopumps, in contrast to the multistage pumps needed to maintain high vacuums required for thermionic cathodes.
As in an ESEM, the MEMSA would include a gaseous secondary-electron detector (GSED) for detecting secondary electrons excited by impingement of the electron beam on the specimen. Electrical charging of an electrically nonconductive specimen by the electron beam would be neutralized by impingement, on the specimen, of positive ions of the residual gas in the chamber. A Positive Intrinsic Negative (PIN) diode would be used as detector for energy-dispersive analysis of $x$ rays generated in the im-
pingement of the electron beam on the specimen.
Unlike in ESEMs and other SEMs, the electron beam would not be rasterscanned across the specimen. Instead, the electron beam would be focused on a fixed spot, through which the specimen would be moved in small steps by use of the translation stage to effect scanning. The amplified output of the GSED acquired at each step would be stored, along with the translation coordinates, in a digital memory, so that an image of the specimen could be reconstructed after completion of the scan. Omitted from the figure for the sake of simplicity is a context imager - essentially, a relatively-low-magnification electronic camera that would facilitate initial coarse positioning of the specimen in or near the electron-beam spot in preparation for scanning.
As in an ESEM, the sample-exchange chamber and the interior volume of the electron source would be separated by pressure-limiting apertures (PLAs), which are small apertures through which the electron beam passes on its way to the specimen. As in an ESEM, the PLAs would be sized to retard the flow of residual gas from the sample-exchange chamber to the electron source side so as to mainain the desired lower pressure


The Micro Electron Probe and Sample Analyzer would function similarly to an environmental scanning electron microscope but would differ in the nature of the electron source and the mode of scanning.
on the electron source side and the desired higher pressure in the sample-exchange chamber. In the original intended application of the MEMSA, the PLAs would be four platinum disks containing apertures of $\approx 0.5-\mathrm{mm}$ diameter, chosen to maintain a pressure between 5 and 7 torr (about 0.67 to 0.93 kPa ) of residual Martian atmospheric gas comprising primarily $\mathrm{CO}_{2}$.

The MEM SA is expected to be capable of imaging at a spatial resolution of 40 nm or finer without EDX, or imaging at somewhat coarser resolution (of the
order of 200 nm ) with EDX in the energy range from 100 to 20 keV . The coarsening of resolution in the case of EDX would be a consequence of the need to use higher electron current. The maximum power demand of the MEMSA during operation has been estimated to be $\approx 5 \mathrm{~W}$.
This work was done by H arish M anohara, Gregory Bearman, Susanne Douglas, Michae Bronikowski, Eduardo U rgiles, and Robert Kowalczyk, of Caltech and Charles Bryson of Apperati, Inc. for NASA's Jet Propulsion Laboratory.

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Innovative Technology A ssets M anagement JPL
M ail Stop 202-233
4800 Oak Grove Drive
Pasadena, CA 91109-8099
E-mail: iaoffice@jpl.nasa.gov
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## (8) N anowire Electron Scattering Spectroscopy

## Multiple chemical compounds could be sensed, without the need to chemically functionalize nanowires.

NASA's Jet Propulsion Laboratory, Pasadena, California

Nanowire electron scattering spectroscopy (NESS) has been proposed as the basis of a class of ultra-small, ultra-low-power sensors that could be used to detect and identify chemical compounds present in extremely small quantities. State-of-the-art nanowire chemical sensors have already been demonstrated to be capable of detecting a variety of compounds in femtomoIar quantities. H owever, to date, chemically specific sensing of molecules using these sensors has required the use of chemically functionalized nanowires with receptors tailored to individual molecules of interest. While potentially effective, this functionalization requires labor-intensive treatment of many nanowires to sense a broad spectrum of molecules. In contrast, NESS would eliminate the need for chemical functionalization of nanowires and would enable the use of the same sensor to detect and identify multiple compounds.

NESS is analogous to Raman spectroscopy, the main difference being that in NESS, one would utilize inelastic scattering of electrons instead of photons to determine molecular vibrational energy levels. More specifically, in NESS, one would exploit inelastic scattering of electrons by low-lying vibrational quantum states of molecules attached to a nanowire or nanotube (see figure). The energy of the electrons is set by the voltage bias applied across the nanowire. When the electron energies correspond to particular molecular vibrational levels, enhanced electronic scattering will lead to a
change in the differential conductance ( $\mathrm{dl} / \mathrm{dV}$, where I is current and V is voltage) at that voltage. Thus changes in the conductance provide a direct readout of molecular vibrational energies, to enable spectroscopic identification of the attached molecules.
To realize a practical chemical sensor based on NESS, one would need a nar-row-energy-band electron source, efficient coupling between the electrons and the molecules of interest, and the narrow vibrational bands in the molecules of interest. A carbon nanotube (CNT) provides a nearly ideal structure for satisfying the electron-source and coupling requirements for the following reasons: Even at room temperature, the energy bands in one dimensional carbon nanotubes are narrow, and low-
energy electrons travel ballistically over distances of the order of a micron, so that injected electrons can have a nearly uniform kinetic energy. Because single-walled CNTs are essentially all "surface," there is strong coupling between electrons and molecules on their surfaces.

O ther than CNTs, nanowires of silicon and perhaps other materials may yield usable NESS signals, though the signals are expected to be smaller than those from CNT-based sensors. One might need non-CNT nanowire NESS sensors to detect molecules that do not readily bind to CNTs.
In order to simplify the interpretation of a complex spectrum from a mixture of compounds, a NESS-based sensor could be integrated with a microfluidic


Inelastic Scattering of Electrons by molecules on the surface of a nanotube would affect the current-versus-voltage characteristic of the nanotube.

