

ROBERT M. FRISTROM, RICHARD C. BENSON, C. BRENT BARGERON,
TERRY E. PHILLIPS, CHARLES E. VEST, C. HOWARD HOSHALL,
R. BEN GIVENS, FRANK G. SATKIEWICZ, and O. MANUEL UY

EROSION STUDIES ON SOLAR MAX SAMPLES

Samples from the Solar Max satellite were retrieved for inspection after more than four years' exposure to the space environment. Efforts were made to identify the most likely erosive processes and responsible species.

INTRODUCTION

The Solar Max satellite, which failed due to malfunction of the inertial reference system after more than four years in orbit, was retrieved and repaired during the third shuttle mission in April 1984 (Fig. 1). The damaged pieces were returned to earth and portions were distributed to interested groups for examination. APL received three samples (shown in Fig. 2): (a) a 110- by 120-millimeter section of a protective blanket (composed of Kapton®, Dacron®, and aluminized Mylar®), (b) a 44- by 47-millimeter section of silver-coated Teflon® that had been shaded from the sun, and (c) a 33- by 54-millimeter section of silver-coated Teflon, portions of which had been exposed to the sun.

The condition of the recovered samples sheds light on the erosiveness of the near-space environment. While a future shuttle experiment is scheduled to test selected samples, the exposure times will be short compared with the Solar Max samples. It was hoped that an examination would provide clues to the identity of the erosive processes and responsible species. Interest centered around oxygen atoms, which are ubiquitous at the satellite orbit altitudes (265-nautical-mile dip orbit) and are known to erode some metal surfaces. We also searched for clues implicating other species.

Our original plans were to compare externally exposed samples with unexposed material and with virgin materials exposed to low-pressure oxygen atoms in the laboratory. A number of tests were completed (Table 1).

DIAGNOSTICS

Three diagnostic methods were used: optical microscopy, scanning electron microscopy, and Fourier transform infrared spectroscopy. Optical microscopy was used to survey the surfaces at low magnifications, and a 5-power magnification mosaic of the samples was prepared. Survey areas were selected for more detailed study and were examined at magnifications up to 400 power using a conventional metallographic microscope. Scanning electron microscopy was performed in an ETEC Autoscan instrument equipped with an X-ray elemental analyzer. Magnifications up to 8000 power were used. Some



Figure 1—An astronaut repairs Solar Max, replacing sections to be returned to earth. (Photograph courtesy of NASA.)

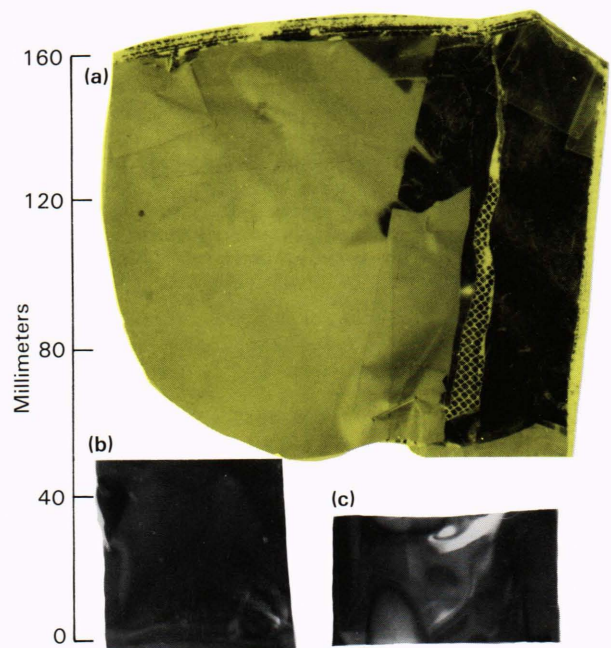


Figure 2—The Solar Max materials provided to APL: (a) the blanket; (b) the unexposed portion; and (c) the exposed portion.

Table 1—The APL program to study returned Solar Max samples.

<i>Sample</i>		<i>Optical Microscopy</i>	<i>Scanning Electron Microscopy</i>	<i>Low-Energy Oxygen-Atom Bombard- ment</i>	<i>Ion Bombard- ment</i>	<i>Fourier Transform Infrared Spectroscopy</i>
Aluminized Kapton	Front etched	C	C			C
	Front impact	C	C			
	Front breathing hole	C	C			
	Front pinhole	C	C			
	Back (overall)	C	C			
Aluminized Mylar	Behind breathing hole	C				
	Overall	C				
Dacron net	Separation net	C				
Silvered Teflon	Cleared area	C	C			
	Boundary	C	C			
Virgin composition	Impacts (sun exposed)	C	C			
	Impacts (unexposed)	C	C	C		
	Kapton	C	C	C	C	C
Virgin composition	Dacron net	C				
	Aluminized Mylar	C				
	Silvered Teflon	C		C		

C = Completed

stereoscopic images were produced from two photographs taken at tilt angles differing by 5 to 6 degrees. Infrared spectra were recorded in the region between 800 and 2200 wavenumbers, where spectral modifications would be expected if the polymers were fundamentally altered.

Two types of simulation were carried out: (a) sample exposure to a flow containing about 2 percent of low-energy oxygen atoms at a pressure of 0.5 torr for 3 hours, and (b) bombardment with 3000-volt argon ions in a secondary ion mass spectrometer.

OBSERVATIONS

Four types of material were examined: aluminized Kapton, Dacron netting, aluminized Mylar, and silver-coated Teflon. The materials were available from both exposed and protected areas.

Aluminized Kapton

The outer skin and backing were of aluminized Kapton with the polymer surface facing outward from the sandwich. The front surface appeared golden because of the Kapton's color; in the virgin state, it had a metallic sheen from the back-surface aluminum mirror. In exposed areas, the Kapton was a dull yellow, while it was mirror bright where the material was protected.

Superficial erosion was examined using scanning electron microscopic pictures under high magnification, up to 8000 power. Scanning electron micrographs of unexposed and exposed Kapton are shown in Fig. 3. The

micrographs suggest a chemical type of etching that produces a wavelike pattern that is presumably due to the inherent microstructure of the polymer. The exposed surface is rather rough, resulting in a dull appearance. Several raised lines were also etched out, suggesting lines of stress in the polymer. Many scratches were observable under backlighting, probably resulting from rough handling at some point. No contaminant elements were detected above atomic number 8, the lower limit of the X-ray system.

The outer Kapton layer of the blanket had an array of holes 3 millimeters in diameter to allow water vapor and trapped gases to escape. In addition to these normal breathing holes, backlighting showed several apparent pinholes. No evidence for pinholes was observed on the underlying Mylar sheet, suggesting that any impacting material became volatile before penetrating the Mylar. The pinholes were about 0.1 millimeter in diameter and were circular. More extensive studies with the scanning electron microscope showed that they were imperfections in the aluminum backing of the Kapton, not impact holes.

Optical and electron microscopic observations of both the Kapton and Teflon samples show a number of other areas that do appear to result from impacts. Examples are shown in Fig. 4. However, none of them shows true penetration or the residue of any element above atomic number 8. Although this does not rule out impacts by ice or carbonaceous particles, it does rule out iron meteorites unless one wishes to postulate a clean hit.

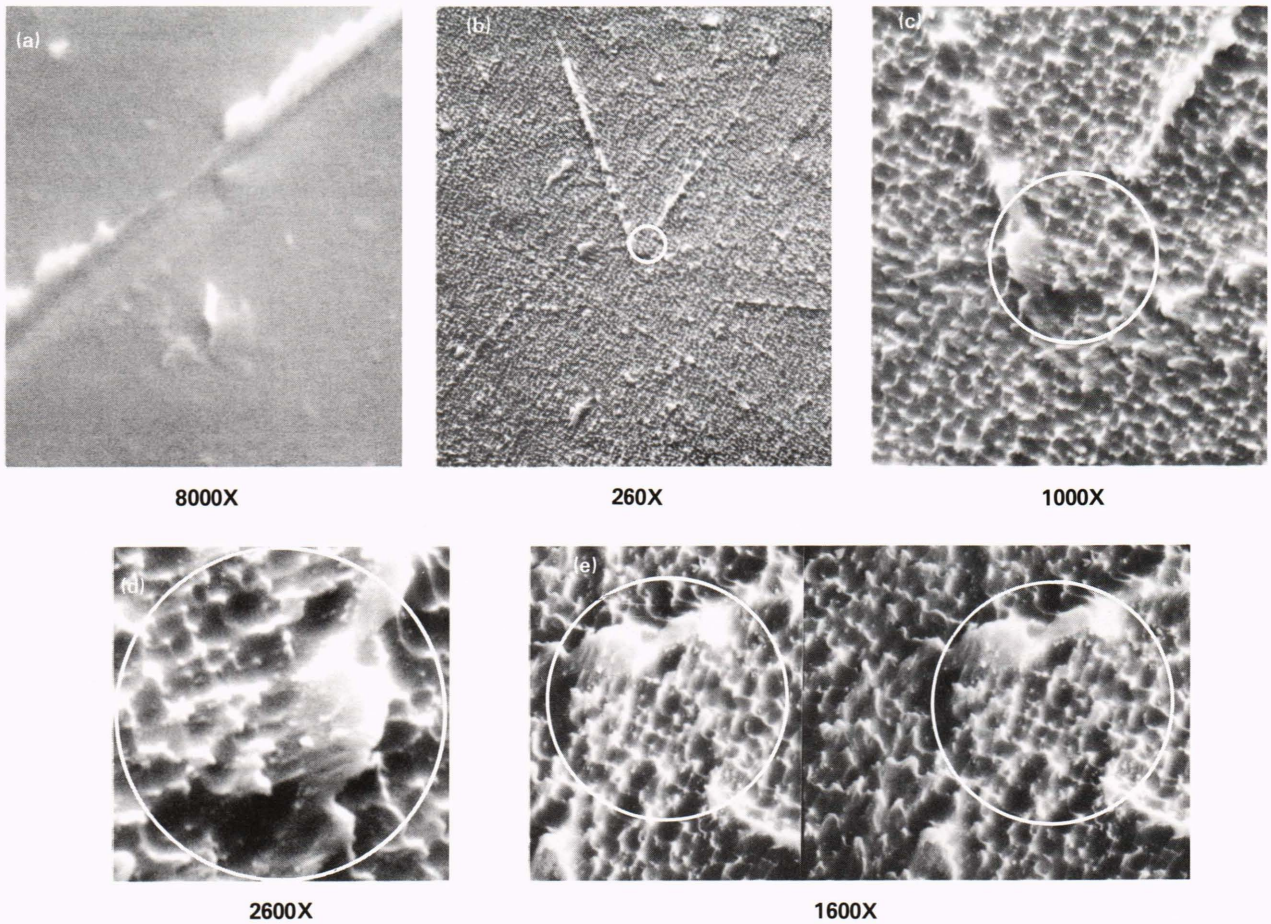


Figure 3—Scanning electron micrographs of unexposed and exposed surfaces of Kapton under various magnifications. (a) The unexposed surface is smooth (having a visually shiny surface) with a few scratches and particulates on the surface. (b), (c), (d) The exposed surface is comparatively roughened, resulting visually in a loss of gloss (matte finish), raised lines, and a few particulates less etched than the surrounding surface. (e) The stereoscopic effect of the pair can be seen by placing a card vertically separating the two images from direct binocular view and relaxing the eyes to bring the image of each eye into coincidence.

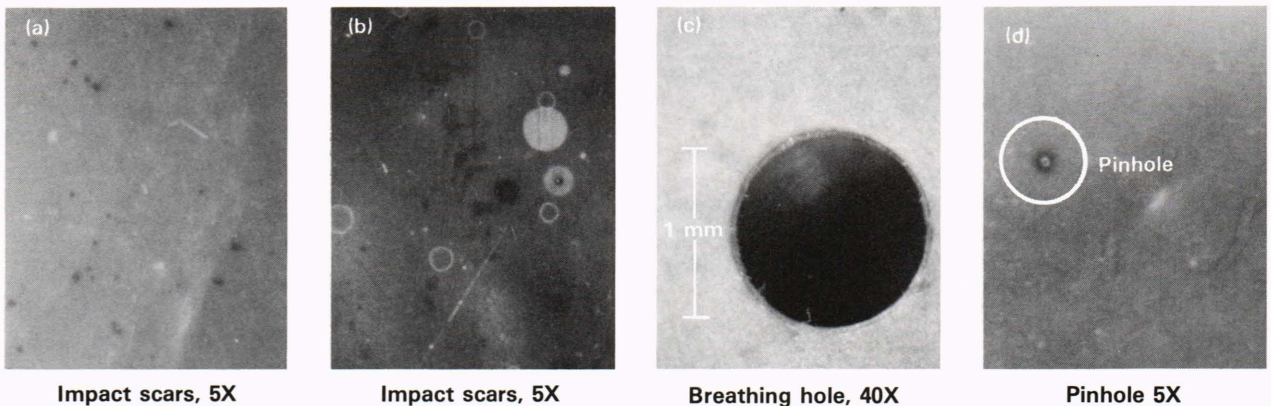


Figure 4—Optical micrographs showing possible impact scars (a and b), a breathing hole (c), and a pinhole (d) in the Kapton blanket.

Fourier transform infrared spectroscopy was performed in the hope of detecting changes in the polymer structure. No significant differences were observed in the exposed Kapton sample as compared with the matched unexposed sample (Fig. 5). Since the method probes the top few microns of the sample, any chemical changes

restricted to the near-surface region (the upper 2 to 3 nanometers) would be obscured by the large signal from the bulk polymer.

Thus, in the case of Kapton, exposure roughens or etches the surface while the residual bulk-polymer structure remains essentially unchanged.

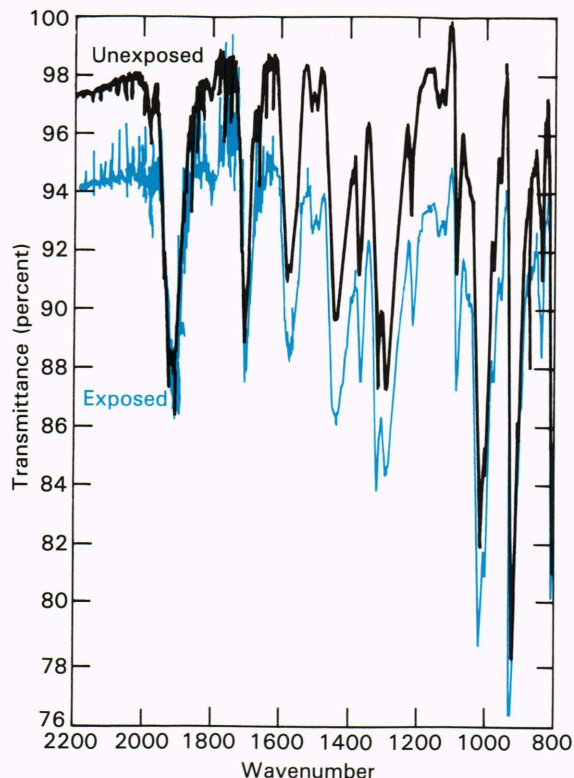


Figure 5—Comparison of Fourier transform infrared spectra of exposed and unexposed Kapton. No significant differences were observed between the two spectra.

Dacron Net and Aluminized Mylar

No etching of the Dacron separation net was observed under optical microscopy. The aluminum-coated Mylar inner films of the blanket were inspected visually using optical microscopy up to 400 power. No erosion was apparent.

Silver-Coated Teflon

The sample of silver-coated Teflon appears to be severely eroded in the area exposed to sunlight. A few islands of silver remain in the eroded area, and there is an increase of island size as the boundary is approached (Fig. 6). Many degradation cracks were found in the illuminated area where the silver coating is not visible (Fig. 7). A small amount of silver remains in this area, as is indicated by the X-ray analysis.

SIMULATIONS

To test the cause of the erosion, we subjected a sample of the unetched Kapton to a low-pressure oxygen-atom discharge. Part of the sample was illuminated by the microwave discharge, which is rich in ultraviolet, and part of the sample was shaded. After a 3-hour exposure, no apparent erosion was observed in either the illuminated or unilluminated portions. To test the possible role of high-energy ions, a sample of virgin Kapton was exposed to a 3-kiloelectronvolt argon-ion beam under high vacuum conditions. Erosion similar to the etching of exposed Solar Max Kapton was observed on this sample.

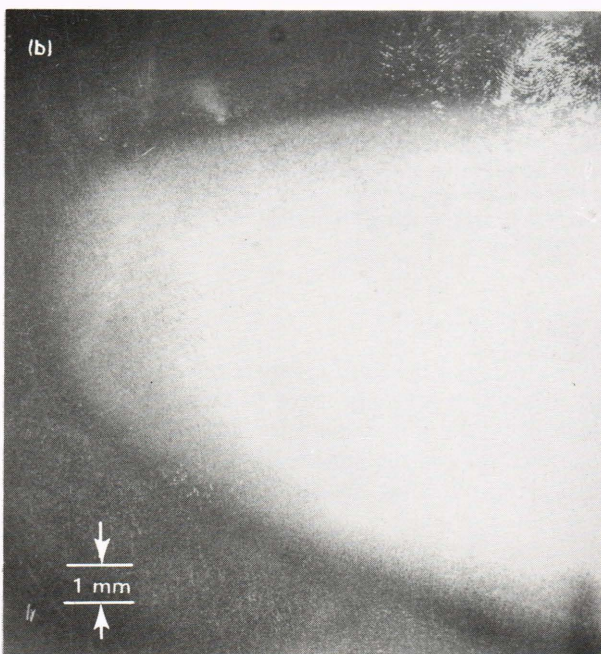
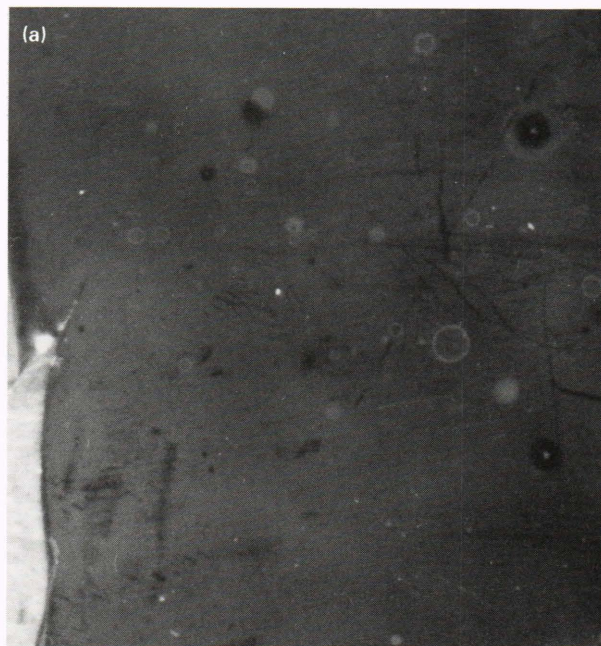


Figure 6—Optical micrographs of silver-coated Teflon showing (a) impact scars and (b) the light-assisted erosion boundary with in which most of the silver has been removed from the surface. The magnification is 5 power for both photographs.

Exposure of the uneroded silver Teflon sheet to a low-pressure oxygen-atom discharge (0.5 torr, 2 percent of low-energy oxygen atoms, room temperature) showed a strong attack on the coating. After 15 minutes, the material took on the appearance of black velvet similar to the “platinum black” of hydrogen electrodes. The attack was more rapid where the material was illuminated by the ultraviolet radiation from the discharge, but even completely shaded material showed some attack after 3 hours. Silver oxide is a gray-black material that, if finely divided, would have a black velvet appearance.



Figure 7—Optical micrograph (30 power) of the eroded silver-coated Teflon surface. According to X-ray analysis, only a small amount of silver remains in the heart of the eroded area. Many degradation cracks are observed in the Teflon in the sun-illuminated area.

CONCLUSIONS

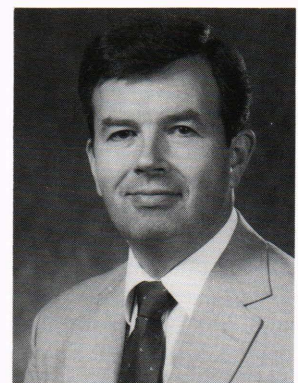
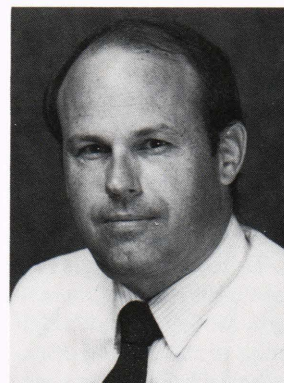
The etching of the Kapton outermost layer is superficial, affecting a layer less than 10 microns deep, as is indicated by the stereoscopic scanning electron microscope pictures and the persistence of 100-micron fibers produced in punching out the breathing holes (Fig. 5).

THE AUTHORS

Robert M. Fristrom (left)

Richard C. Benson (center)

C. Brent Barger (right)



Further evidence for the surface attack is the lack of any change in the infrared signature of the bulk polymer.

The Kapton etching probably requires high-energy species. This is suggested by our failure to reproduce the etching using room-temperature oxygen atoms at an equivalent exposure. However, we should be cautious about assigning the etching to a single species. It is suggestive that even the chemically inert argon ions give a similar etching at sufficiently high acceleration energies. Among the possible candidate species are H, O, N, OH, O₂ (excited), O₃, NO⁺, O⁻, Ar⁺, and N₂⁺.

Another candidate for causing erosion was the sub-micron micrometeorite population, which could be large if the log-normal distribution continues down to molecular levels instead of peaking in the micron-size region, as is assumed at present.

Low-energy oxygen atoms are apparently not a factor in the Kapton erosion at the altitude of the Solar Max orbit because oxygen atoms in the laboratory showed no signs of attacking. By contrast, oxygen atoms strongly attacked the silvered Teflon but in a different way from that observed on the exposed satellite material, which was only eroded in the areas exposed to light.

Both aluminized Mylar and the Dacron net materials of the blanket were resistant to the agent that etched the Kapton, an observation that is substantiated by the absence of visible attack on these materials in the area under the breathing hole, where they were exposed to the same environment as was the Kapton.

Although there is evidence suggesting micrometeorite impacts, they were neither large nor energetic enough to penetrate the Kapton blanket and left no residue of elements above atomic number 8. This suggests that the micrometeorites were either icy or carbonaceous.

ACKNOWLEDGMENTS—This work was supported by the Naval Sea Systems Command under Contract N00024-85-C-5301. We would like to thank the NASA Satellite Servicing Project and G. W. Ousley, Jr., for furnishing the samples and coordinating the information exchange.

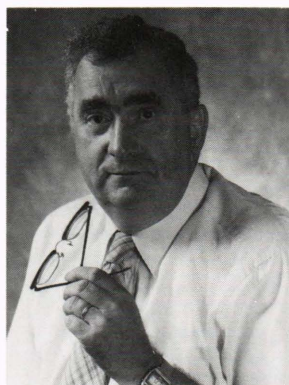
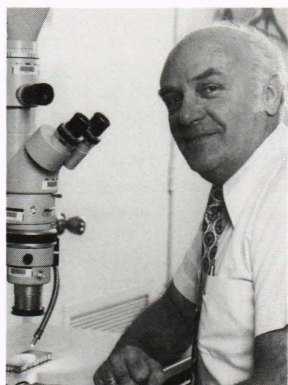
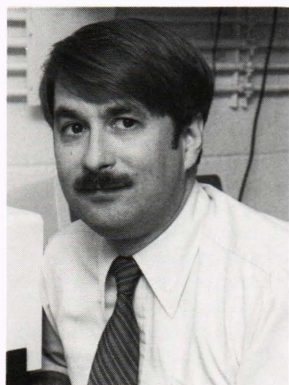
ROBERT M. FRISTROM was born in Portland, Ore., and received degrees in chemistry from Reed College (B.A.), the University of Oregon (M.A.), and Stanford University (Ph.D.). He was a Research Fellow in chemistry at Harvard University from 1948 to 1951. In 1951, he joined APL in the Bumblebee Propulsion Group. Presently he is in the Computational Physics Group of the Milton S. Eisenhower Research Center. His interests at APL have included propulsion, microwave spectroscopy, flames, fire, molecular beams, and chemical kinetics.

Dr. Fristrom has been awarded the Parsons Fellowship, the Silver Medal of the Combustion Institute, the Hillebrand Award of the Chemical Society of Washington, the Certificate of Achievement from the Armed Services Explosives Safety Board, and one of the first Humboldt Foundation awards (of West Germany) to a U.S. scientist. His publications include three books and over 90 technical and review articles.

RICHARD C. BENSON was born in 1944 and was educated at Michigan State University (B.S. in physical chemistry, 1966) and the University of Illinois (Ph.D. in physical chemistry, 1972). Since

joining APL in 1972, he has been a member of the Milton S. Eisenhower Research Center and is manager of the Surface Science Program. He is currently involved in research on the properties of materials used in microelectronics and the application of optical techniques to surface science. Dr. Benson has also conducted research in Raman scattering, optical switching, laser-induced chemistry, chemical lasers, energy transfer, chemiluminescence, fluorescence, and microwave spectroscopy. He is a member of the American Physical Society and the American Vacuum Society.

C. BRENT BARGERON joined APL in 1971 as a member of the Milton S. Eisenhower Research Center. Born in Provo, Utah, in 1943, he earned a Ph.D. degree in physics at the University of Illinois (1971). His thesis was on high-pressure physics. Since joining APL, Dr. Bargeron has been involved in problems in solid-state physics, light scattering, chemical lasers, arterial geometry, corneal damage from infrared radiation, mineral deposits in pathological tissue, quality control and failure analysis of microelectronic components, electron physics, and surface science.



Terry E. Phillips (left)
Charles E. Vest (center)
C. Howard Hoshall (right)

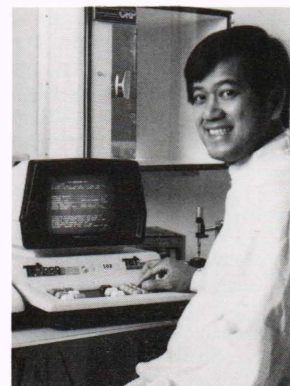
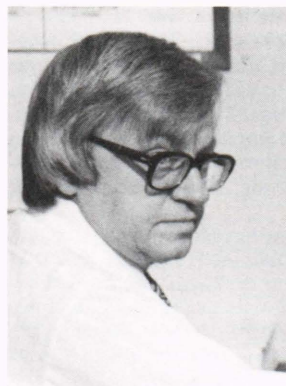
TERRY E. PHILLIPS is a senior staff chemist in the Materials Science Group in the Milton S. Eisenhower Research Center. Born in Sunbury, Pa., he received a B.A. from Susquehanna University and M.S. (1972) and Ph.D. (1976) degrees in chemistry from The Johns Hopkins University. After completing postgraduate studies at Northwestern University in low-dimensional organic conductors, he joined APL in 1979. Dr. Phillips has been involved in problems in photoelectrochemical energy conversion, inorganic optical and electrical phase-transition compounds and materials characterization with X rays, nuclear magnetic resonance, mass spectroscopy, and optical spectroscopic techniques.

CHARLES E. VEST received a B.S. in metallurgical engineering from the Virginia Polytechnic Institute (1950) and an M.S. in materials engineering from the University of Maryland (1967). His professional experience includes research on nuclear metallurgy, refractory metals metallurgy, spacecraft materials, reactions of the spacecraft environment with materials, materials test and analysis equipment and procedures, and metallographic technologies. Mr Vest joined APL in 1983 after 25 years with NASA and the Atomic Energy Commission and 10 years in related industries. He is a materials

engineer and manager of the Technical Services Department's Materials Laboratory. He has been involved with materials evaluations in the HUT, POLAR BEAR, Delta 180, Delta 181, and other space programs. He has also worked on materials problems for other departmental programs at APL.

C. HOWARD HOSHALL was born in upper Baltimore County, Md., in 1926 and received a B.S. degree in electrical engineering from the University of Maryland in 1949. He was employed for several years in government and industry before coming to APL in 1962. At APL, Mr. Hoshall has had a wide variety of assignments involving electronic systems, instrumentation, and hardware design. Among them were early assignments in the Space Department, development of electrically powered upper-extremity prostheses and orthoses, and laboratory investigations to determine how ultrasound might be used to detect presymptomatic atherosclerotic lesions in major arteries. Since 1977, he has been engaged in the development of special-purpose apparatus and instrumentation in the Combustion Research and Neutral Beam Laboratories in the Milton S. Eisenhower Research Center.

R. Ben Givens (left)
Frank G. Satkiewicz (center)
O. Manuel Uy (right)



R. BEN GIVENS was born in Huntington, W. Va., in 1933. He graduated from the Army Electronic School in 1955 and Capitol Radio Engineering Institute in 1957. He then joined Litton Industries, where he was responsible for the performance of their instrument landing systems. Mr. Givens joined the Milton S. Eisenhower Research Center in 1965 and since 1983 has been modifying the scanning electron microscope to operate as a scanning electron acoustic microscope.

FRANK G. SATKIEWICZ is a member of the Materials Science Group of the Milton S. Eisenhower Research Center. A native of Cambridge, Mass., he earned degrees in chemistry at Northeastern University (B.S.), Wesleyan University (M.A.), and M.I.T. (Ph.D.). Prior to his Ph.D studies, he was a radiochemist at Tracerlab, Inc., and a high school teacher. Subsequently, he was employed at Norton Co. doing research on abrasives, refractories, and zeolites. He later worked on the principal staff of GCA Corp. in developing materials for space applications and as a consultant in using sputter-ion source mass spectrometry for studying solids. Since joining APL in 1973, Dr. Satkiewicz has concentrated on using and improving secondary ion mass spectrometry for the analysis of solids, and re-

cently has been conducting experiments on the acoustical response of metals when subjected to an ion beam.

O. MANUEL UY, a member of APL's Principal Professional Staff, is supervisor of the Quality Assurance Section of the Space Reliability Group. Born in Cadiz City, Philippines, he studied at La Salle College, Manila, where he graduated with a B.S. in chemistry and chemical engineering in 1962. He came to the United States on a Fulbright scholarship and studied at Case Western Reserve University, where he received his Ph.D. degree in chemistry in 1967. He specialized in mass spectrometry and high-temperature chemistry as a Welsh Postdoctoral Fellow at Rice University in Houston and as a NATO Postdoctoral Fellow at the Free University of Brussels. Dr. Uy was a senior scientist at Space Sciences, Inc., where he studied rocket and flame additives, and was laboratory chief at Cleveland's Air Pollution Control Division. He later worked at G.E.'s High Intensity Discharge Lamp Department, as senior engineer, unit supervisor, and product evaluation manager. He joined APL in 1981 as a specialist in quality assurance.

Dr. Uy is a member of the American Chemical Society, the Royal Society of Chemistry, Sigma Xi, the American Society of Mass Spectrometry, and the American Institute of Aeronautics and Astronautics.