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Results from the Space Technology Research Vehicle 1a Atomic Oxygen Experiment

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An experiment to make in situ measurements of thermospheric atomic oxygen fluxes and their effect on some common spacecraft materials has been flown in orbit on board the Space Technology Research Vehicle 1a microsatellite. The results from active sensors were based on resistance measurements of silver films and were interpreted relative to space simulations made in a ground-based pulsed laser atomic oxygen source. It was concluded that silver resistance sensors can be used to measure atomic oxygen fluxes but are best suited to low flux environments, e.g., $<10^{14}$ atoms cm^{-2} s^{-1}. Some silver sensors were overlaid with thin coatings to measure the erosion resistance of the coating materials. It was found that erosion rates were affected by the structure of the materials; this structure must therefore be taken into account in experiment and spacecraft design.

Introduction

SPACECRAFT in low Earth orbit (LEO) travel through the thermosphere, the region of the Earth's neutral atmosphere between altitudes of approximately 90 and 1000 km. The predominant constituent of this environment is atomic oxygen (AO), produced by the photodissociation of molecular oxygen. The AO concentration at a particular altitude depends on solar heating and thus the level of solar insolation. Spacecraft in LEO may therefore encounter AO density variations of one or two orders of magnitude due to seasonal and diurnal heating variations and the solar cycle.

Although these AO concentrations are very low, the high velocity of a LEO spacecraft causes large amounts to be swept onto forward-facing surfaces. AO is highly reactive, and this flux causes the chemical erosion of many materials commonly used on spacecraft, leading to degradation in their performance and the possibility of equipment failures.

Samples of space materials returned during the Apollo and Skylab programs were frequently affected by outgassing contamination so that AO effects were not observable.[1] With the early Space Shuttle missions, however, when significant quantities of space-exposed materials could be returned to Earth undamaged by re-entry, the implications of the AO erosion of spacecraft structures were fully realized.[2,3] Since then, a variety of space- and ground-based experiments have investigated orbital AO concentrations and their effects on materials.

Laboratory simulations obviously offer a high degree of access at a lower cost than space experiments, but because of problems in reproducing the space environment, particularly the production of AO beams with orbital velocities (~8 km s^{-1}) at realistic flux levels, there tends to be a lack of confidence in the results of this ground-based research.

Orbital experiments are therefore more desirable, but because of the need to return materials to Earth after exposure, these experiments, with the notable exceptions of the Long Duration Exposure Facility[4] and European Retrievable Carrier[5,6] spacecraft, have been limited to Space Shuttle[7–10] and orbital station[11,12] missions. To make accessible a wider variety of orbital conditions and to reduce the cost of flight experimentation, a simple orbital instrument capable of making in situ measurements of both AO flux and material erosion rate has been developed. The first demonstration of this instrument was the Atomic Oxygen Experiment (AOE), launched on the Space Technology Research Vehicle 1a (STRV-1a) spacecraft in June 1994 (Ref. 13).

Experiment

In-orbit atomic oxygen experiments have previously been conducted using various techniques, including mass spectrometers,[14,15] quartz crystal microbalances,[16,17] catalytic probes,[18,19] and silver film actinometers.[20] For the AOE, a design based on silver films was developed because it offered the best possibility of meeting the objectives of both measuring AO flux and coating erosion rates in a controlled-temperature environment while meeting the stringent experiment mass and power budgets allowed by the STRV-1a microsatellite. These budgets resulted in an experiment mass of 360 g and a peak power consumption of 2.3 W. Because of a satellite battery problem, use of the temperature-controlling heater was curtailed early in the mission, after which the peak power consumption was 1.2 W.

Silver film actinometers were first used in space on sounding rocket experiments[21–23] and more recently have been employed on both retrievable[24] and nonretrievable[25] orbital spacecraft. In this study more emphasis was placed on the use of silver to measure AO fluxes and the role of silver film thickness on sensor sensitivity.

The principle of the sensor is as follows: On exposure to AO a silver film will react to form electrically nonconducting silver oxides. The progress of this reaction can be monitored remotely by measuring the film's resistance, measurements that can be used to estimate the AO flux. Interpretation of resistance data must take into account film thicknesses, substrate surface roughness, contact resistances, and temperature.[26]

To measure the erosion rates of materials, coatings of these materials were overlaid on some of the AOE sensors. When coatings were breached by AO erosion an increase in the resistance of the underlying silver films was observed. Very thin silver films were used for coated sensors so that the resistance change was immediately apparent. Knowledge of the AO fluence experienced up to these points and the coating thicknesses enabled the erosion rates of the overlays to be calculated.

The AOE comprised 12 sensors mounted in a 5 × 10 cm space on the outside of the STRV-1a spacecraft. Each sensor consisted of a thin silver film of dimensions 12 × 0.25 mm, configured in a shallow U shape to conserve space, vacuum evaporated onto a ~1-cm^2 glass substrate. For the coated sensors the polymer and silica overlays were deposited onto a 10 × 7 mm area covering the silver films through ion beam sputtering a target of bulk material. The carbon overlays were deposited onto a similar area via the arc evaporation of carbon electrodes. Details of the sensors and the overlay materials flown are given in Table 1.

A thermofoil heater below the sensor substrates was used to keep the temperature of the sensors constant to protect the mechanical

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Table 1 AOE silver film sensors and overlays

Sensor ID	Silver thickness, Å	Coating	Coating thickness
FA	1840	——	——
FB	1870	——	——
FI	2840	——	——
FJ	2790	——	——
FE	190	PTFE	950 Å
FF	270	PTFE	950 Å
FK	320	PE	1.15 μm
FL	240	PE	1.15 μm
FG	270	Carbon	1600 Å
FH	280	Carbon	1600 Å
FC	330	SiO_2	200 Å
FD	340	SiO_2	200 Å

integrity of the films and to allow AO interactions and resistance measurements to be made at a controlled temperature. Leads from the sensors were connected to a measurement unit inside the spacecraft. A full description of the experiment's design, construction, and operation is given in Ref. 13.

STRV-1a was one of a pair of 50-kg microsatellites launched into a 7-deg-inclination geostationary transfer orbit (GTO) on June 17, 1994. The spacecraft was spin stabilized; the rotation rate was ~5 rpm with the spin axis approximately perpendicular to the direction of motion.

The highly elliptical GTO is an unusual orbit for AO experimentation as significant AO fluxes are experienced for only the short period of each orbit (in this case ~15 min of each 10.5-h orbit) when the spacecraft approaches and leaves perigee. However, the high rate of the silver/AO reaction means that uncoated silver actinometers have short lifetimes in LEO.[24] GTO was therefore advantageous as the low AO fluence per orbit extended the life of the experiment, resulting in improvements in accuracy as well as easing data gathering and storage requirements. However, due to the higher perigee velocity (~10 km s^{-1}), the AO impact energy was higher than that encountered in LEO (7.7 and 4.5 eV, respectively, allowing for the corotation of the equatorial atmosphere). The literature indicates no energy dependence of the reaction rate of silver with AO in this range, and so the increase is unlikely to have affected the silver film reaction rates.[27,28] It may, however, have affected the erosion yields of some of the polymer coatings used on the AOE. In addition, the effect of the altitude variation of AO density had to be taken into account in the analysis and interpretation of results.

During the first week of the STRV-1a mission, the temperature of the sensors was constrained to $25 \pm 2^{\circ}$C using the thermofoil heater. Unfortunately, the subsequent failure of a spacecraft battery cell interrupted the vehicle's operating schedule. After resumption of normal operations, spacecraft power conservation procedures meant that it was not possible to use the AOE heater. Temperature readings from the AOE indicated a lowest sensor temperature of -11°C, experienced during the short eclipse that initially occurred just before perigee. Although this is not thought to have influenced results from the uncoated silver sensors, it is possible that results from the overlays, which may have more temperature sensitive reaction rates, e.g., carbon, may have been affected.

Ground-based testing in support of the AOE was conducted in the European Space Research and Technology Centre ATOX simulation facility. This facility uses an AO generation technique based on a pulsed laser-induced breakdown method to produce a thermally cold AO beam with a hyperthermal velocity.[29] The velocity of the AO beam during these tests varied in the range 8–9 km s^{-1}, giving AO energies of 5.3–6.6 eV.

Laboratory work included tests that simulated both typical Space Shuttle fluxes (circular LEO, altitude ~250 km) of $\sim 5 \times 10^{15}$ atoms cm^{-2} s^{-1}, for comparison with the literature, and the mean GTO flux anticipated for STRV-1a ($\sim 3 \times 10^{14}$ atoms cm^{-2} s^{-1}). Kapton H witness samples were mounted with the sensors for quantitative mass loss experiments, and bulk samples of the sensor coating materials were also tested.

Results and Discussion

Uncoated Sensors

Measurements of the orbital AO flux environment were made using four uncoated silver film sensors (Table 1). Figure 1 shows that the resistance of sensors FA and FB, both ~1850 Å thick, increased to 50 Ω (the limit of measurement) after 280 and 330 orbits, respectively. The resistance of sensors FI and FJ, both ~2800 Å thick, remained approximately constant during the lifetime of the experiment (~700 orbits; the orbital period was constant). The small resistance decrease at 300 orbits corresponds to the resolution of the measurement equipment and was probably caused by a gradual change in other environmental conditions, such as a decreasing operating temperature.

Data from some laboratory exposures of equivalent bare silver sensors are shown in Figs. 2 and 3. Figure 2 shows data from an exposure simulating Space Shuttle orbital conditions, whereas Fig. 3 shows data taken when flux conditions were more typical of GTO.

Comparison of the flight experiment sensors FA and FB and the laboratory simulation tests shows that the resistance-vs-time curves had similar forms. Initially there was a high rate, linear resistance increase, followed by a low rate, parabolic stage and finally a catastrophic tertiary stage. The latter was associated with the final breakup of the conduction path in the silver films and was therefore ignored in subsequent quantitative analysis.

The resistance measurements were converted into silver loss values using an empirical relationship based on a theoretical consideration of conduction in thin metallic films.[13,26] Silver loss results based on Figs. 2 and 3 are shown in Figs. 4 and 5, respectively. The slopes of the data from sensors FA and FB during the initial linear period of rapid silver loss, which existed until approximately 350 Å of silver was converted to oxide, were matched to those of

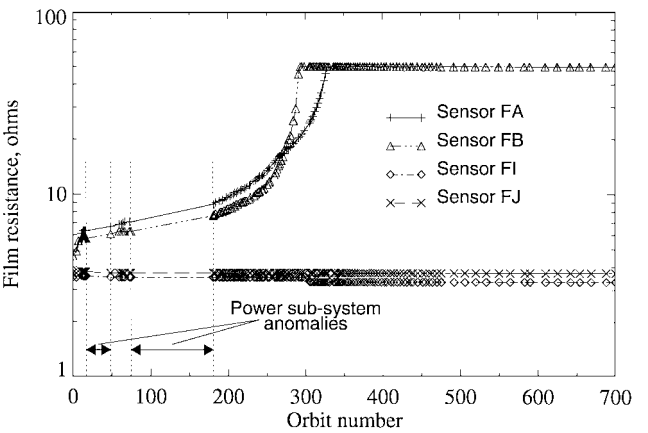


Fig. 1 Temperature-corrected resistance vs orbit number for the uncoated AOE sensors.

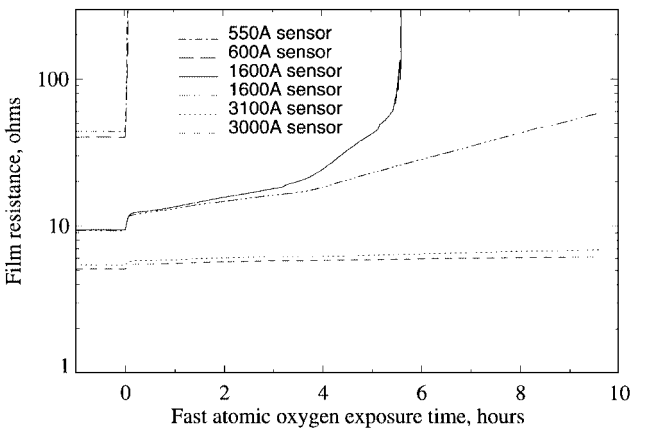


Fig. 2 Resistance vs time data for uncoated sensors exposed in ground-simulation tests to AO fluxes typical of Space Shuttle orbital altitudes ($\sim 5 \times 10^{15}$ atoms cm^{-2} s^{-1}).

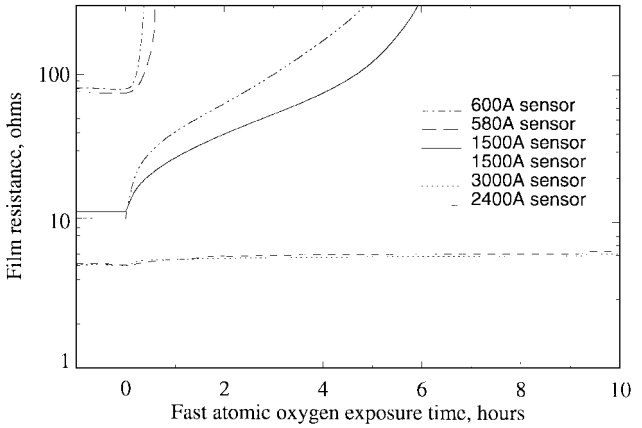


Fig. 3 Resistance-vs-time data for uncoated sensors exposed in ground-simulation tests to AO fluxes close to the mean flux expected for the STRV-1a GTO ($\sim 3 \times 10^{14}$ atoms cm^{-2} s^{-1}).

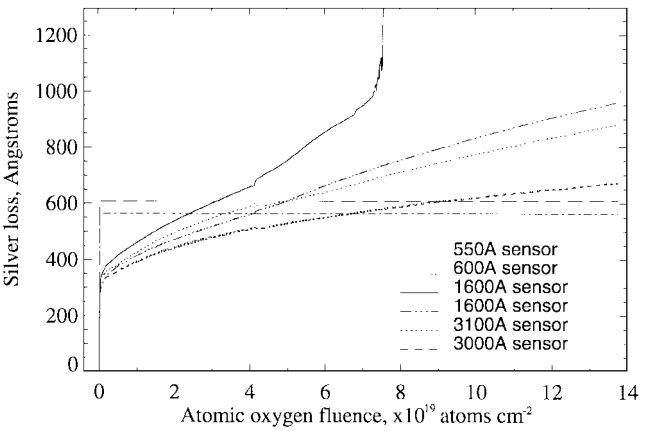


Fig. 4 Silver lost vs AO fluence for the uncoated sensors portrayed in Fig. 2 (exposed to conditions typical of Space Shuttle altitudes).

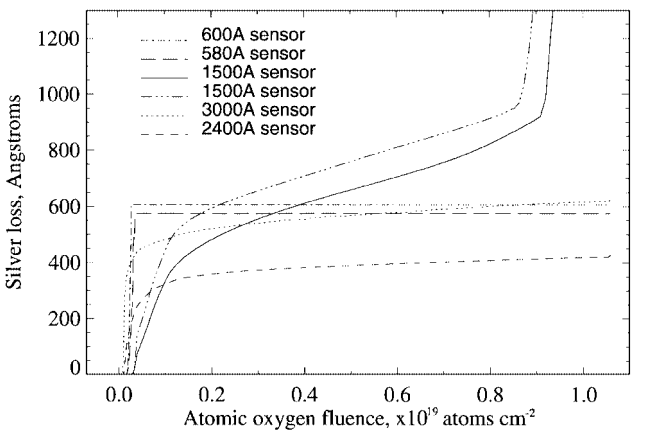


Fig. 5 Silver lost vs AO fluence for the uncoated sensors portrayed in Fig. 3 (exposed to conditions close to the mean flux expected for the STRV-1a GTO).

the laboratory data using a multiplying factor (Fig. 6). This factor was found to be 2×10^{16} atoms cm^{-2} orbit^{-1} and can be taken as the estimate of the AO flux per STRV-1a orbit during the early orbit phase. For the same period and orbit, the ESABASE thermospheric AO model predicted a flux of 5.6×10^{16} atoms cm^{-2} orbit^{-1}. Given the uncertainties in both values, this relative agreement is encouraging, although until further measurements are obtained, it must be viewed with caution.

The laboratory results showed differences between higher and lower flux conditions. An example of this flux dependency is shown in Fig. 7, which includes close-ups of two films from Fig. 4. On two occasions the AO flux was increased; the frequency annotations in

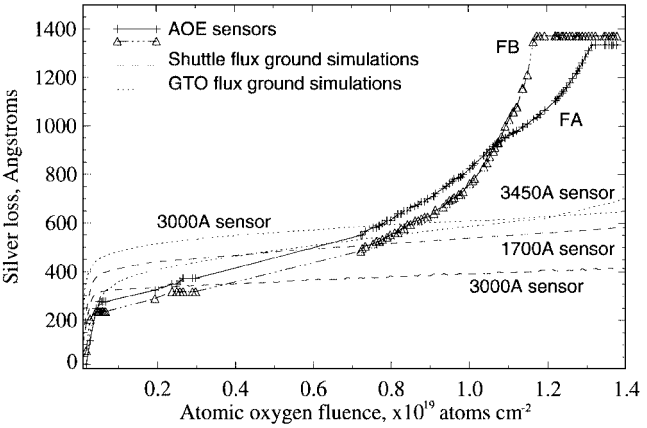


Fig. 6 Comparison of silver loss from AOE uncoated sensors FA and FB and ground simulations assuming a constant perigee AO flux for the AOE.

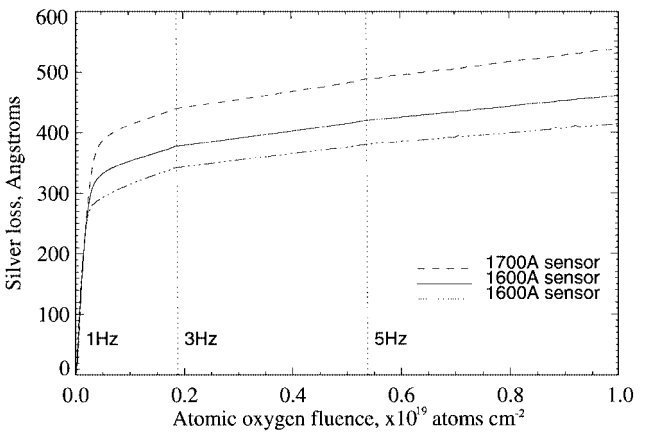


Fig. 7 Silver loss vs AO fluence for several sensors exposed in ground-based simulations, showing the flux dependence of the silver film reaction rate. The frequency annotations indicate increases in the pulse rate of the ATOX laser and relate directly to the AO flux generated.

Fig. 7 refer to the pulse rate of the ATOX facility laser and directly relate to the relative AO flux. The reaction rate (with respect to time) of the silver films increased by a smaller factor than that of the AO flux, corresponding to a decrease in the per atom reaction rate. This can be explained by considering that at high fluxes the films' surfaces are saturated by AO and excess AO does not therefore increase the oxygen concentration and the diffusion-limited reaction process that depends on it.

Although the linear regions of the AOE and ATOX results can be matched, the later flight data diverge from the laboratory data, indicating an increased silver film erosion rate in orbit (Fig. 6). Factors such as oxide flaking due to fatigue induced by orbital conditions, e.g., thermal cycling and vibration, could account for these observations, although no sharp resistance changes, indicative of large-scale oxide flaking, were observed. A more likely explanation is the increase in AO flux as a result of the known reduction of STRV-1a's perigee height, from 280 to 220 km, during this phase of the mission. However, in a pulsed AO facility, such as the one used for laboratory testing here, the instantaneous AO flux can be several times higher than the mean flux. Diffusion limiting of the laboratory reaction may therefore have resulted in the per atom reaction rate being higher than was calculated. This effect would also contribute to the divergence observed in Fig. 6.

To investigate further, the ESABASE model was used to estimate per orbit fluxes based on the spacecraft's measured orbital ephemerides, and values for the cumulative AO fluence at various times during the first five months ($\sim$350 orbits) of the mission were calculated. Data from films FA and FB were replotted using this fluence record and compared with the laboratory data (Fig. 8). This figure shows the flight data matching the LEO flux laboratory data later in the mission. This supports the view that the decrease in the

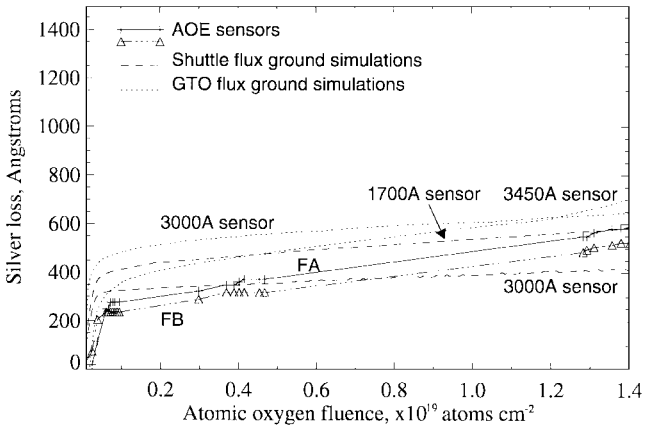


Fig. 8 Comparison of silver loss from AOE uncoated sensors FA and FB and ground simulations assuming a decreasing perigee/increasing AO flux for the AOE.

STRV-1a perigee was the main reason for the divergence between laboratory and flight data, although a contribution from the flux dependence of the laboratory reaction rates in the parabolic stage is likely.

Given the reactions of AOE films FA and FB, some response would have been expected from the thicker uncoated silver sensors FI and FJ. Based on the AO fluence measured by FA and FB during their lifetimes, a resistance increase in the range 2–4.5 Ω would have been expected from FI and FJ over the same period. This resistance change is well above the sensitivity of the instrument (0.2 Ω). Both surface contamination of the sensors and local electronic circuit failures are possible explanations of these results. The latter is thought to be less likely because analysis of other output from the AOE measurement electronics suggested that the sensors were operational for the full lifetime of the mission. In contrast, the danger of surface contamination is a recurrent feature of most AO erosion experimentation. For the AOE, sensors FA and FB were manufactured together and placed in adjacent locations on the outside of STRV-1a. FI and FJ were also manufactured together, but separately from FA and FB, and were located ~8 cm from FA and FB. Each pair of sensors therefore experienced slightly different manufacturing, handling, and ambient conditions, which may explain contamination differences.

Coated Sensors

The resistance of polytetrafluoroethylene (PTFE)-coated sensor FF started to increase after 180 orbits, and it rapidly rose to 1000 Ω (the limit of measurement). The other PTFE-coated sensor, FE, went open circuit during a second STRV-1a power anomaly, which occurred between orbits 75 and 180. (During this period contact with the satellite was lost and no data were acquired.) Based on an AO fluence estimate provided by ESABASE for the period of its operation (1.3×10^{19} atoms cm^{-2}), the erosion rate of film FF was 0.72×10^{-24} cm^3 $atom^{-1}$. Because of the uncertainty in the time that the resistance of film FE started to increase, the erosion rate of this coating can only be stated to be within the range $0.77–2.0 \times 10^{-24}$ cm^3 $atom^{-1}$. These values are similar to those obtained for coated sensors exposed in the ATOX facility ($0.6–1.9 \times 10^{-24}$ cm^3 $atom^{-1}$). Such values, obtained with both thin film overlays and bulk samples in the laboratory, are over twice as high as the best values previously measured in flight experiments, e.g., $0.20–0.37 \times 10^{-24}$ cm^3 $atom^{-1}$ (Refs. 30 and 31).

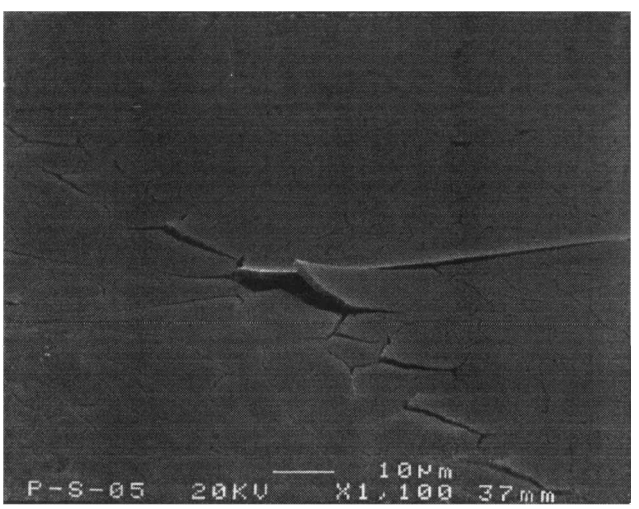
There are two possible explanations of these observations. One concerns the role of vacuum ultraviolet (VUV) radiation in the degradation process of PTFE. The flight sensors experienced high levels of VUV radiation both simultaneously with AO exposure (during perigee) and alone (during most of the rest of the orbit). Similar VUV radiation is also produced by the ATOX source.[32] It has previously been suggested that VUV and possibly other parts of the electromagnetic spectrum play an important role in the degradation behavior of fluorinated polymers by contributing energy to bond-breaking processes.[33–35]

The exact role of short-wavelength radiation is, however, unresolved. An alternative explanation is that the higher AO energies of GTO accelerated the erosion rate. The carbon-hydrogen bonds found in hydrocarbons, such as polyethylene (PE), have a bond energy of 4.24 eV, i.e., lower than the energy imparted by an orbital AO collision. In contrast, the carbon-fluorine bonds found in PTFE and other fluorocarbons have an energy of 5.02 eV. This may be high enough to inhibit AO erosion, particularly in low-inclination, prograde orbits. In the STRV-1a GTO and in the ground testing here, typical AO energies were higher (7.7 and 5.3–6.6 eV, respectively) and may have promoted the PTFE/AO reaction.

No resistance changes were observed for the PE-coated sensors, implying that they did not fully erode during the lifetime of the mission. Calculations based on AO fluences predicted by ESABASE suggest that the films should have eroded through after approximately 400 orbits. As with the uncoated sensors FI and FJ, contamination and electronic circuit failures are again possible explanations, although scanning electron microscope examinations of the laboratory-exposed samples offer an alternative explanation.

Examinations of laboratory-exposed PE coatings and bulk samples revealed differences in the erosion morphology. The bulk samples exhibited the classic rug-like texture,[36] whereas the thin sputtered overlays were free from the expected erosion pattern but were extensively cracked (Fig. 9). Thus, in the latter case, it is likely

a) Bulk PE exposed to 1.4×10^{20} atoms cm^{-2}; in the center is a debris particle that has shielded the underlying surface from erosion (oblique view, magnification ×2500)



b) Cracks in a thin film PE coating exposed to 0.28×10^{20} atoms cm^{-2} (oblique view, magnification ×1100)

Fig. 9 Scanning electron micrographs of PE exposed to fast AO in ground-simulation tests.

that the laboratory sensors were measuring degradation of the overlays due to cracking rather than to surface recession. The results strongly suggest that material degradation as a result of exposure to AO erosion or a combined AO and VUV environment is dependent on material structure and method of manufacture. Factors such as crystallinity, surface finish, deposition parameters, and bond orientation have been found to influence degradation rates and morphologies in polymers.[37,38]

Hence, with regard to the PE-coated flight sensors, whether resistance increases occur may well depend on whether the overlay films crack during the life of the experiment. This in turn will depend on their method of manufacture, which was identical to the laboratory sensors, and the combined orbital environment (AO, VUV, and thermal). This argument applies to all measurement techniques that use materials in a form that is not identical to that used in service. For design purposes, "lifeing calculations" must be based on appropriate samples and orbital environments.

No resistance change was observed for the carbon-coated sensors, either in the flight experiment or in the laboratory AOE simulation. Work published since the launch of the AOE and some of the ground-based simulations described here indicate a strong, direct temperature dependency of the reaction rate of hyperthermal AO and carbon.[27] The thickness chosen for the carbon overlays was probably too great to fully erode under the AO dose experienced by the AOE and in the ATOX facility at ambient temperatures, a problem compounded by the curtailment of heater use in the case of the flight experiment.

Silica films, which were expected to be protective, were also flown on the AOE and tested in ground-based simulations. The resistance of both silica-coated overlay sensors slowly increased just before the termination of the flight experiment, suggesting a gradual failure of the protective layers. The films exposed in the laboratory were still protective after exposure to an equivalent AO fluence. Some work has suggested pinholes, introduced during manufacture, as a reason for the inability of silica, as well as some other potentially protective materials, to protect against oxygen attack.[39] Interdiffusion between the silica and silver films has also been suggested.[40] A further explanation is also the mismatch between the thermal properties of silica and silver. Thermal fatigue failure in the stable environment of the laboratory exposures would be much less likely than in the thermally unstable orbital conditions.

Evaluation of the Experiment

A major objective of the AOE was to demonstrate that silver resistance sensors, without overlays, could be used to remotely monitor the orbital AO environment over a period of several weeks. In this respect the experiment was a success; realistic flux measurements were derived from the observations, and there was close agreement with thermospheric model estimates.

However, the experiment also had limitations. It is clear from this study that only the first-stage linear oxidation is sufficiently sensitive and reproducible to be of use in flux measurement. Because this stage is short lived (up to a silver loss of 350 Å), the technique is most suited to elliptical orbits such as GTO or high Earth orbit where AO fluxes are relatively low. Unless the sensors are protected from ram exposure to AO during the launch phase, the technique is not readily applicable to higher flux orbits, such as LEO.

Surface contamination of the sensors was the main problem that affected the AOE, and although this experiment was based on silver film sensors, this problem equally applies to other techniques that rely on chemical reactions for observables. In laboratory simulations only 3% of films failed to respond, whereas for the space flight experiment two of the four uncoated films failed. A burn-in period has been reported by other workers,[21,28] probably due to the removal of organic contamination or a silver sulfide layer, the latter formed before tests due to the action of ambient sulfur dioxide, and this was observed to a small degree in laboratory exposures here, e.g., Fig. 5. With thin films, any burn-in period is well defined because the resistance of the silver starts to increase rapidly once the contamination is etched away. However, with thicker films this is not the case as the initial rate of resistance increase from oxidation is low due to the films' lower overall resistance.

Because the initial, linear part of the silver/AO reaction regime is the most reliable for estimating fluxes, the thickness of the silver films is important. If they are too thick, e.g., >3000 Å, they may be too insensitive to produce precise measurements. If they are too thin, e.g., <300 Å, they incur the risk of being unstable. The experience of the AOE indicates that film thicknesses in the range 600–2000 Å represent the best compromise, the exact value depending on the anticipated AO environment.

Although only four of the eight coated sensors provided definite results during the lifetime of the experiment, the technique of using the resistance increase of a thin silver film to determine when an overlay coating has failed is believed to be sound. However, it should be remembered that it provides a measure of the resistance to the orbital environment of material with a specific structure, in this case sputter coatings. This structure may not necessarily be representative of the material in its bulk form. The PE results of this study tend to support this argument. There may also be effects associated with the uneven failure of the coating, which should be further investigated and characterized.

Conclusions

An investigation of thermospheric atomic oxygen fluxes and their degradation effects on some common spacecraft coatings has been performed using a suite of thin silver film sensors carried by the STRV-1a microsatellite. In low-flux environments, the sensors provided reasonable measurements of the AO flux, a value of 2×10^{16} atoms cm^{-2} $orbit^{-1}$ being obtained for a GTO in the period June 1994. For the same period and orbit, a thermospheric model gave a value of 5.6×10^{16} atoms cm^{-2} $orbit^{-1}$. For the analysis used here, film thicknesses in the range 600–2000 Å are believed to be optimal.

Resistance increases of sensors overcoated with test materials indicated erosion yields for PTFE coatings comparable to those of some laboratory experiments, i.e., $0.7–2.0 \times 10^{-24}$ cm^3 $atom^{-1}$, although higher than in most experiments conducted by other groups. This enhancement may be due to simultaneous AO and short-wavelength solar radiation exposure or to the higher AO collision energy encountered in elliptical GTO. The material samples were in thin film form, and results indicate that further work is required to characterize the influence of material structure and to quantify errors resulting from overlay breakdown end effects. The main drawback of the technique is the difficulty of avoiding surface contamination of sensors, although it proved possible to reduce this risk and to obtain useful measurements.

Acknowledgments

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