

Low Earth Orbit Durability of Protected Silicone for Refractive Photovoltaic Concentrator Arrays

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Photovoltaic power systems with novel refractive silicone solar concentrators are being developed for use in low Earth orbit (LEO). Because of the vulnerability of silicones to atomic oxygen and ultraviolet radiation, these lenses are coated with a multilayer metal oxide protective coating. The objective of this work was to evaluate the effects of atomic oxygen and thermal exposures on multilayer coated silicone. Samples were exposed to high-fluence ground-laboratory and low-fluence in-space atomic oxygen. Ground testing resulted in decreases in both total and specular transmittance, while in-space exposure resulted in only small decreases in specular transmittance. A contamination film, attributed to exposed silicone at coating crack sites, was found to cause transmittance decreases during ground testing. Propagation of coating cracks was found to be the result of sample heating during exposure. The potential for silicone exposure, with the resulting degradation of optical properties from silicone contamination, indicates that this multilayer coated silicone is not durable for LEO space applications where thermal exposures will cause coating crack development and propagation.

Introduction

SINCE 1986, NASA Lewis Research Center has been developing the minidome Fresnel lens photovoltaic concentrator. This novel, point-focus refractive concentrator space power system offers extremely high power density, relatively low weight, radiation hardness, and potential for array cost reduction compared to existing systems.^{1–3} The minidome Fresnel-lens concentrator uses a unique optical design (a domed shape with individually tailored Fresnel facets on the inside surface) that is very efficient and shape error tolerant. Figure 1 is an illustration of a single square-edged minidome Fresnel lens. The general concept of the point-focus minidome concentrator has also been applied to a linear-focus design. The linear Fresnel-lens concentrator has the same advantages as the point-focus design with the added advantage of requiring precise sun tracking only within a single axis.⁴ These advanced refractive concentrator designs, along with the introduction of new high-efficiency photovoltaic devices, could have significant use in future power systems for a wide range of missions, including low earth orbit (LEO), geosynchronous orbits, and high-radiation orbits.^{1–4}

Under the current advanced refractive concentrator program, the concentrator lens is made using a flexible silicone. Because silicones are known to react with atomic oxygen,⁵ the predominant species in the LEO environment,⁶ and to darken with ultraviolet radiation exposure, a multilayer metal oxide coating to protect against atomic oxygen (AO) and ultraviolet (UV) radiation is deposited onto the silicone exterior. This coated silicone configuration is being considered as an alternative to earlier ones in which the lens is covered with ceria-doped microglass.^{1–3}

The efficiency of a refractive photovoltaic concentrator system is directly related to the ability of the lens to focus incident radiation onto the active area of the solar cell. Therefore, the con-

centrator lens must maintain a high solar specular transmittance. The purpose of this investigation was to evaluate the LEO durability of protected silicone for refractive concentrator systems. The primary concerns are differences in the coefficient of thermal expansion (CTE) between the silicone substrate and the metal oxide protective coating, atomic-oxygen interaction with exposed silicone at coating defect sites with potential silicone contamination, and the overall protection afforded by the multilayer coating. This evaluation was conducted with both ground-laboratory and in-space exposures.

Materials and Experimental Procedures

Materials

The materials evaluated were flat DC 93-500 silicone, coated with a proprietary 11-layer ($\text{SiO}_2/\text{Al}_2\text{O}_3/\text{Ta}_2\text{O}_5$) thin-film AO- and UV-resistant coating, and uncoated DC 93-500 silicone. The 11-layer coating was deposited by plasma-enhanced sputter deposition. Flat material was evaluated instead of refractive lenses, so that optical properties could be measured.

Characterization of Optical Properties

Total, diffuse, and specular transmittances were obtained using a Perkin-Elmer λ -9 spectrophotometer equipped with a 60-mm integrating sphere. Solar integrated values were obtained by measuring the spectral transmittance over the wavelengths of 250–2500 nm and convoluting the data into the air-mass zero-solar-intensity curve over this range. The spectral transmittance uncertainty is $\pm 2\%$.

Surface and Mass Characterization

Changes in surface morphology as a function of AO fluence were observed using optical microscopy, scanning electron microscopy (SEM), and atomic force microscopy (AFM). The optical microscope used was an Olympus SZH stereo microscope, and micrographs were obtained at magnifications between 50 and 95 times. A Cambridge 200 scanning electron microscope was used for high-magnification imaging (up to 13,000 times). Samples were coated with conductive Au films prior to SEM examination. AFM analysis was performed using a Park Scientific AutoProbe. X-ray photoelectron spectroscopy (XPS) analysis was conducted on pristine and ground-laboratory AO-exposed uncoated DC 93-500. Both XPS surface analysis and XPS depth profiling were conducted. Dehydrated

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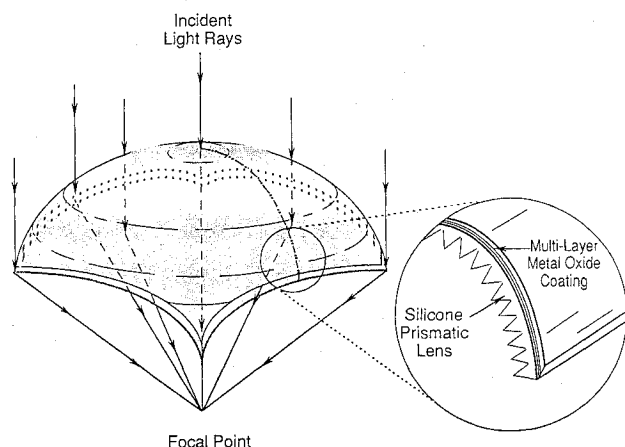


Fig. 1 Minidome Fresnel lens photovoltaic concentrator: before and after severe bending and before and after flight.

mass measurements were obtained using a Sartorius balance, with an uncertainty of $\pm 50 \mu\text{g}$.

Ground-Laboratory Atomic Oxygen

A Structure Probe Inc. plasma asher was used to produce an AO plasma environment. In addition to AO, the plasma contains a strong 130-nm line of UV radiation.⁷ Although the plasma asher does not provide identical AO exposure conditions to those in the LEO environment (because of differences in energy, flux, arrival direction, species, etc.), ashers are generally accepted as ground-laboratory systems for atomic-oxygen durability evaluation. A plasma asher provides an inexpensive and timely way to conduct ground-laboratory AO exposure compared to other sources of AO, such as AO beam facilities, which are expensive, are not always accessible, and generally provide a low AO flux. Effective fluence exposures were based on the average of many flux measurements obtained by ashing Kapton witness coupons in the asher prior to exposure of silicones, and calculating the flux from the mass loss of the Kapton and the known erosion yield of Kapton in LEO.⁸ This has been shown to be an acceptable method of estimating the expected flux of a given asher exposure.

One drawback to using this method is that the rate of AO reaction in the asher is dependent on the temperature of the sample. The higher the temperature, the faster the reaction rate. Because this method of estimating fluences does not take into account the difference in temperature between the Kapton witness coupons and the actual samples, the effective fluences stated in this paper should be taken as first-order approximations. Samples were exposed to estimated effective fluences of up to 2.6×10^{21} atoms/cm² based on a flux of 3×10^{15} atoms/cm² s.

One large and six small multilayer metal-oxide-coated DC 93-500 samples were exposed to ground-laboratory AO. The large sample (sample G7) was used for optical property measurements and was protected on the uncoated side with Al foil so that only degradation to the coated surface would occur and be analyzed. Sample G7 was iteratively exposed to an effective AO fluence of 2.6×10^{21} atoms/cm². Optical properties and dehydrated mass measurements were obtained every 48 h. Five small samples (samples G2, G3, G4, G5, and G6) were placed on glass slides in the asher with sample G7. One small sample was removed every 48 h for destructive SEM evaluation. A sixth small sample (sample G1) was kept unexposed. Each successive small sample for SEM evaluation had effective fluences ranging from 0 to 2.6×10^{21} atoms/cm² in increments of 5.2×10^{20} atoms/cm².

LEO Atomic Oxygen

One uncoated flight sample (F1) and one multilayer-coated DC 93-500 flight sample (F2) were exposed to the LEO environment on the Environmental Oxygen Interaction with Materials (EOIM-III) Experiment. This experiment was exposed to LEO direct ram atomic oxygen on STS-46 and received an approximate fluence of 2.3×10^{20} atoms/cm². Samples exposed in space received direct

ram atomic oxygen exposure to the front side only and were set on polished stainless-steel disk holders during flight. Thermocouples attached to the underside of an aluminized Kapton sample in the same EOIM-III tray indicate that the tray temperature cycled between the temperatures of ≈ 15 and 45°C .

Results

Surface Characterization Prior to Atomic Oxygen Exposure

The pristine multilayer metal-oxide-coated DC 93-500 samples contained coating cracks prior to any environmental exposure. These cracks are attributed to the effects of sample heating during deposition and are due to CTE mismatches between the coating and the silicone as well as flexure during handling. Uncoated silicone samples did not contain any surface cracks prior to AO exposure.

Ground-Laboratory Atomic Oxygen

The total transmittance of sample G7 was found to decrease from 0.834 to 0.796 (a decrease of 0.038) after a fluence of 1.0×10^{21} atoms/cm², and then to remain stable around 0.793 until the final fluence of 2.6×10^{21} atoms/cm² (see Fig. 2). The specular transmittance was found to decrease continuously with exposure from 0.749 to 0.606, a decrease of 0.143 (also shown in Fig. 2). Interference fringe patterns were observed in the spectral data for both the total and specular transmittance scans. Figure 3 shows an interference pattern in the total transmittance spectra between 350 and 1200 nm, which developed during AO exposure. A single maximum (≈ 550 nm) and minimum (≈ 460 nm) of the interference pattern were first observed after a fluence of 1.0×10^{21} atoms/cm². This interference pattern continued to develop throughout exposure, extending to larger wavelengths. The development of the interference pattern is likely to be the result of the deposition of a thin film on the surface of the sample. Silicone contamination of the Kapton witness coupon was observed during initial exposure. Space-flight exposures such

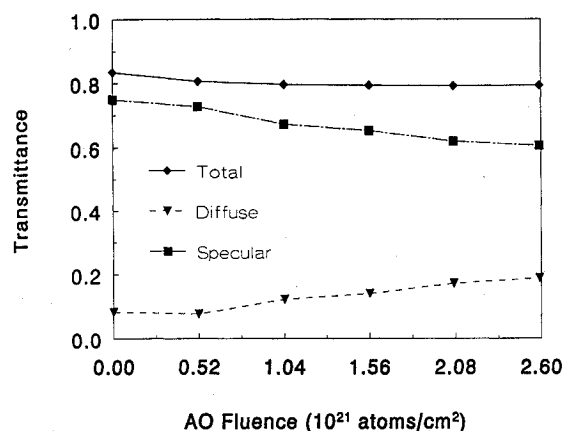


Fig. 2 Transmittance changes as a function of AO fluence for sample G7.

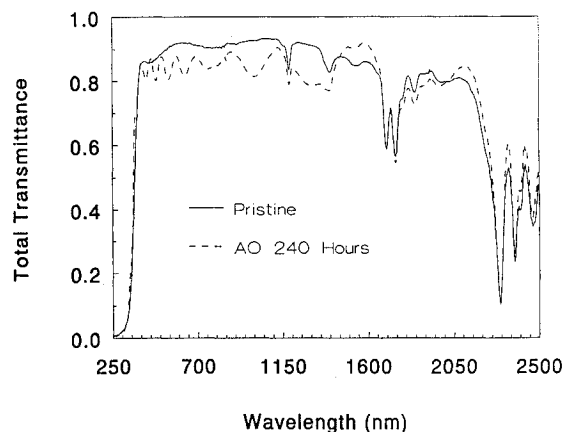


Fig. 3 Total transmittance spectra of sample G7 before and after AO exposure.

as on the Long Duration Exposure Facility (LDEF) have provided evidence of spacecraft silicone contamination with exposure to a vacuum environment containing AO and UV radiation.^{5,9} The interference pattern therefore appears to be the result of cross-contamination from silicone at the coating crack sites, which reacted with AO (in a UV-containing vacuum environment), resulting in a contaminant layer on the metal oxide coating. One of the LEO durability concerns with coated silicone advanced refractive concentrators is the possibility of cross-contamination from the silicone. These results indicate that some initial total and specular transmittance loss are due to silicone contamination. Because sample G7 was ashed along with samples G2–G6, the edges of samples G2–G6 could have been additional sources of silicone contamination products. Therefore the transmittance decrease observed during ground-based exposure may be somewhat accelerated.

Optical microscopy of sample G7 revealed the development of additional coating cracks with continued AO exposure (see Fig. 4). The extent of additional coating cracking with plasma asher exposure was found to be very area specific. Figure 4 shows the area before and after AO plasma exposure, with the most severe additional cracking observed. Note the nonuniform cracking density in Fig. 4c after a fluence of 2.1×10^{21} atoms/cm². Cracking associated with AO

plasma exposure was found to continue to progress with increased exposure time. This is likely to be due to the thermal cycling, that was induced during the iterative ashing exposure. Refractive concentrators in space would also experience thermal cycling. The development of coating cracks will result in decreased specular transmittance.

Additional experiments were conducted to determine if the additional cracking of the multilayer coating during ground-laboratory exposure was due to AO interactions or due to heating, which can occur during plasma ashing (particularly with metals). Using adhesively attached temperature indicators, it was found that coated silicone samples were exposed to temperatures above 116°C in the plasma environment when they were completely protected with Al foil, and to approximately 49°C when the samples were not in contact with metal (i.e., wrapped in polymer material). Both temperatures were found to cause equivalent amounts of additional cracking of the protective coating. The cracking observed is attributed to the differences in coefficient of thermal expansion between the silicone and the metal oxide thin films ($\approx 10^{-4}$ and $\approx 10^{-6}^\circ\text{C}^{-1}$, respectively). Figure 5 shows additional cracking of a coated silicone sample (G8) after being totally encased in Kapton during plasma ashing. Encasing the sample in Kapton resulted in sample heating, with no AO exposure. This sample was exposed for 42 h (equivalent to a fluence of 4.5×10^{20} atoms/cm²). When a sample was encased in a Kapton/Al envelope, placed on a small water-cooled holder in a heated asher, and maintained at 18°C for 48 h, the amount of additional cracking was significantly decreased, with the few additional cracks being attributed to handling.

It was originally estimated that during LEO operation, the minidome Fresnel lenses would be exposed to temperatures up to $\approx 40.5^\circ\text{C}$. A coated sample (sample G9), placed in a regular furnace maintained at $\approx 40.5^\circ\text{C}$, was found to show an increase in coating cracks after a single 48-h exposure (see Fig. 6). This cracking intensity is almost as severe as the cracking observed with sample G7 after a fluence of 5.2×10^{20} atoms/cm². Most of the crack formation observed during iterative AO asher exposures is thus attributed to heating rather than AO interaction, and operating temperatures in

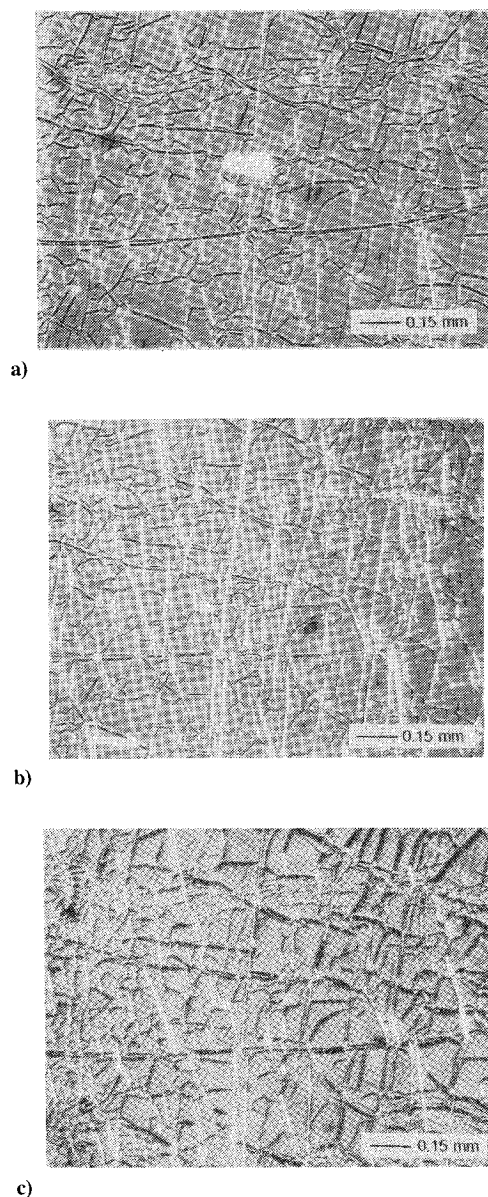


Fig. 4 Development of additional coating cracks on sample G7 with AO plasma exposure: a) pristine, b) AO $F = 5.2 \times 10^{20}$ atoms/cm², and c) AO $F = 2.1 \times 10^{21}$ atoms/cm².

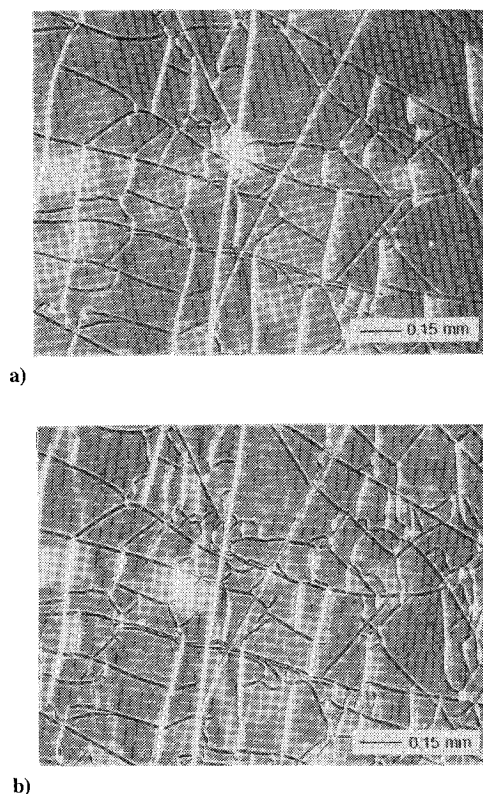


Fig. 5 Additional coating cracking of a coated silicone sample (G8) after being totally encased in Kapton during plasma ashing ($\approx 49^\circ\text{C}$): a) prior to exposure and b) after exposure.

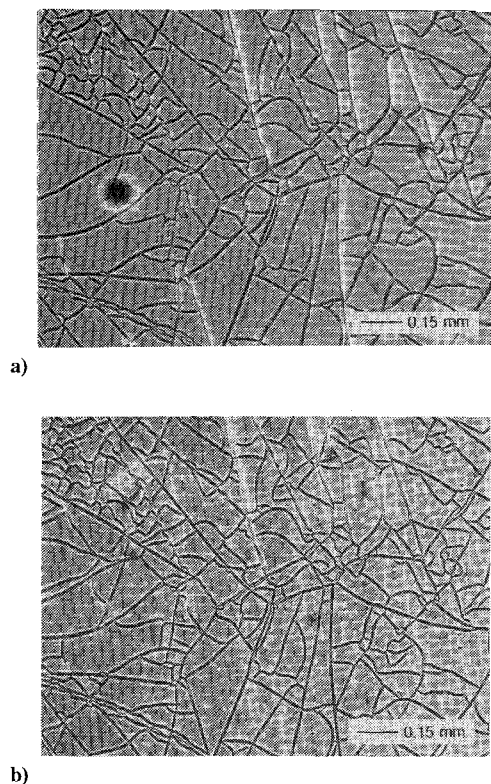


Fig. 6 Additional cracking of a coated silicone sample (G9) after being placed in a 40.5° furnace for 48 h: a) prior to exposure and b) after exposure.

space as low as 40°C will cause significant cracking of the protective coating.

Tests were performed to evaluate the effect of additional cracking on the optical properties. An unexposed coated sample (sample B1) was characterized for transmittance, and optical micrographs were taken. This sample was bent in half, then rolled along the bend line several times. Optical microscopy revealed extreme additional cracking of the coating after bending and rolling, as can be seen in Fig. 7. The postbend optical measurements revealed decreases in total and specular transmittance by only 0.005 and 0.023, respectively. Therefore, any decreases in total and specular transmittance in excess of these values should not be attributed to protective-coating cracking. Transmittance decreases that are not associated with additional cracking are attributed to film growth on the coated surface, as indicated by the presence of the interference pattern in the spectral data, and are likely due to cross-contamination from the exposed silicone at fresh crack sites (and the unprotected sides of samples G2–G6).

In addition to the changes in transmittance of sample G7, there was a gain in mass of 0.46% with AO exposure (see Fig. 8). Scanning electron microscopy of samples G2–G6 showed the apparent formation of a smooth glassy layer on the exposed silicone in coating crack sites with AO exposure. The mass-gain and microscopy analysis provided evidence suggesting the formation of an SiO_x layer on the exposed silicone areas. To confirm this silicone-to- SiO_x conversion process, XPS analysis was performed on four uncoated DC 93-500 samples. Two of these samples were pristine, and two were exposed to AO in a plasma asher with effective fluences of 6.11×10^{19} and 4.12×10^{20} atoms/cm². The oxygen concentration on the surface of these samples showed a marked increase from ≈ 31 to ≈ 59 atomic percent (A%), after an AO fluence of 4.12×10^{20} atoms/cm², while the percentage of silicon remained constant. The majority of oxygen concentration increase occurred after an AO fluence of 6.11×10^{19} atoms/cm² (≈ 57 A%), indicating that the rate of oxygen increase slowed with exposure time (see Fig. 9). Further analysis of these samples revealed that the percentage of SiO_x on the surface of the samples increased from $\approx 26\%$ to $\approx 77\%$ after a fluence of 4.12×10^{20} atoms/cm². The majority of the SiO_x conversion

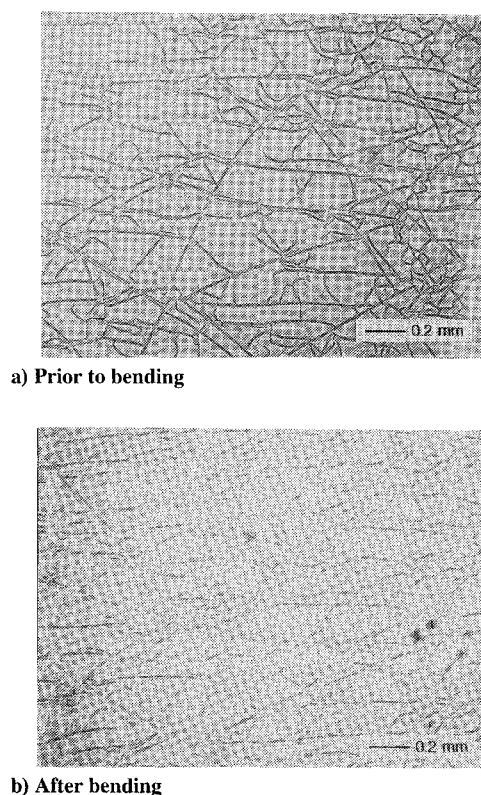


Fig. 7 Effect of severe bending (sample B1) on additional coating cracks.

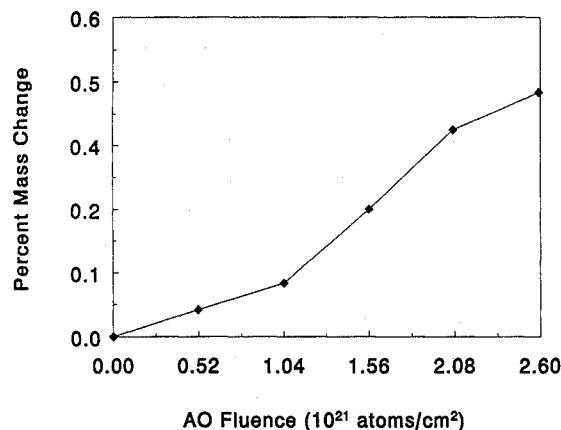


Fig. 8 Gain in mass of sample G7 with AO plasma exposure.

occurred within a fluence of 6.11×10^{19} atoms/cm² ($\approx 72\%$). The decrease in the rate of this conversion process with longer fluences is attributed to less surface area of the silicone being exposed to the AO as the silicate layer forms. Depth profiling showed that the SiO_x layer increased in depth as the AO effective fluence increased. At 6.11×10^{19} atoms/cm² the depth of the SiO_x layer was measured to be ≈ 1000 Å, and at 4.12×10^{20} atoms/cm² it was measured to be greater than 2000 Å. The continued conversion of silicone to SiO_x after an initial surface SiO_x layer has formed, is speculated to be due to the ability of AO to diffuse through the SiO_x layer.

When the surface of silicone converts to SiO_x with AO exposure, it typically loses organic side groups such as methyl groups (note the loss of carbon in Fig. 9), shrinks, and causes mud-tile-like cracks. SEM evaluation of samples G2–G6 provided images where silicone in the coating cracks had converted to SiO_x , cracked, formed SiO_x in that crack, and cracked again. This type of SiO_x conversion cracking propagation is shown in Fig. 10. Figure 10a shows a freshly opened crack with a stretched appearance (vertical) and an older crack that has a smoothed-out, glassy appearance (horizontal). Figure 10b shows cracking of the converted SiO_x layer inside a coat-

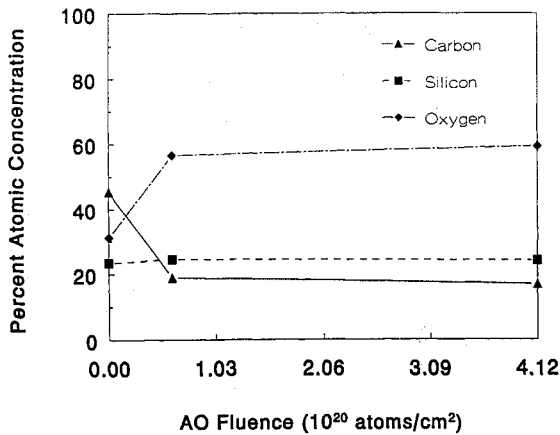


Fig. 9 Surface atomic concentrations of O, C, and Si for uncoated DC 93-500 silicone as a function of AO fluence.

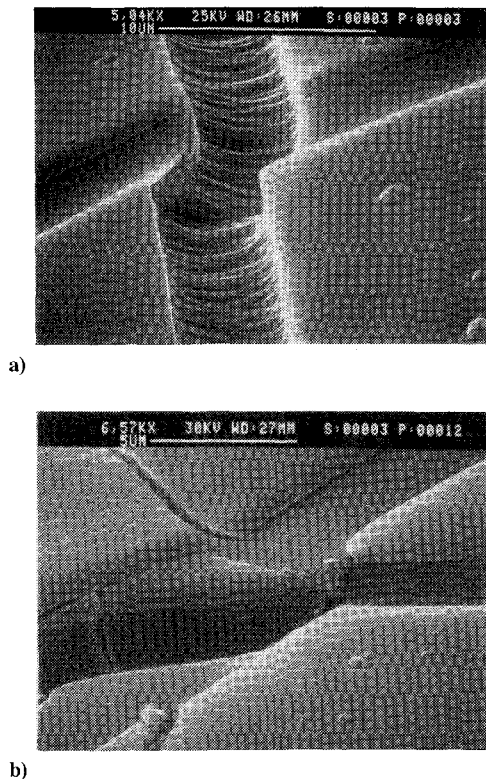


Fig. 10 SEM micrographs showing the development of coating cracks after an AO fluence of 2.6×10^{21} atoms/cm²: a) a freshly opened crack (stretched appearance), and an older SiO_x converted crack (smooth, glassy appearance); and b) cracking of SiO_x converted film inside a protective coating crack.

ing crack. "Bulging" of the coated areas between cracks was also observed with SEM. Typically this bulging was found to increase with increased AO exposure. This effect is observed as extra shadowing in the higher-fluence optical micrographs, such as the one in Fig. 4c. In some isolated areas the bulged coating appears to have partially delaminated from the silicone substrate, so that the edges are free and lifted up, as can be seen in Fig. 11. Only a few coating islands were actually found to have flaked off. The scattering of incident light by these bulges could be a possible source of specular transmittance loss. SEM evaluation also provided evidence that the multilayer coating remained protective and did not appear eroded by AO in the areas where it was not cracked.

LEO Atomic Oxygen

Sample F2, the coated DC 93-500 sample, exposed to directed ram space AO, experienced essentially no change in the integrated total transmittance and a 0.031 decrease in the integrated specular trans-

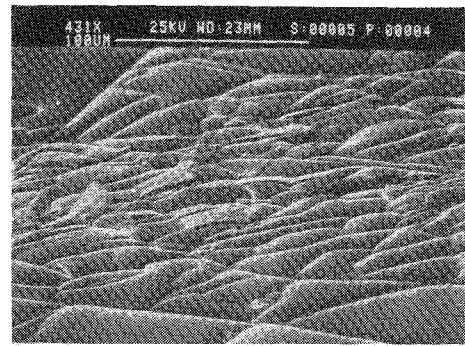
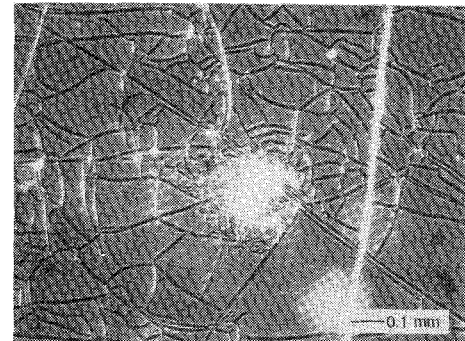
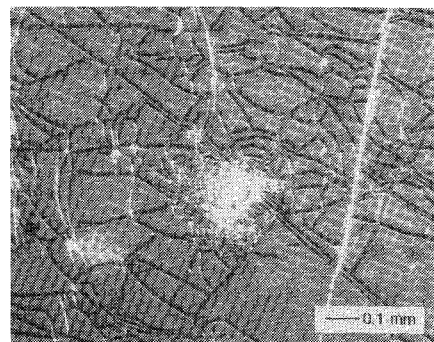


Fig. 11 Isolated area of partially delaminated bulged coating islands.



a)



b)

Fig. 12 Coated DC 93-500 silicone exposed to LEO on STS46: a) before flight and b) after flight ($F = 2.3 \times 10^{20}$ atoms/cm²).

mittance. This combination is consistent with the changes observed in the bend-test sample. However, optical postflight macrographs of sample F2 show only a slight amount of additional cracking (see Fig. 12), not enough to account for the specular transmittance change according to results from the bend-test sample (the coating defect at the center of the macrograph was purposely put there prior to flight). Atomic-force-microscope topography profiles have shown similar bulging of coated silicone sample F2 to that observed with the ground-laboratory samples (see Fig. 13). Scattering of light by these bulges may account for additional specular transmittance losses in excess of those due to cracking. The cracking is probably due to heating and thermal cycling of the sample while in orbit. As previously mentioned, the tray in which this sample was placed cycled between temperatures of ≈ 15 and 45°C . These temperatures have been shown to be sufficient to cause cracking of the protective coating.

Postflight optical and atomic-force microscopy of the uncoated silicone sample F1 showed a cracked surface layer. This coating is the result of conversion of silicone to SiO_x with AO exposure (see Fig. 14). Small changes were noted in both the total and specular transmittance spectra of sample F2. These changes are not consistent with the changes that occurred in total or specular transmittance spectra of the bend-test sample. The bend-test sample shows virtually no change in total transmittance spectra before and after

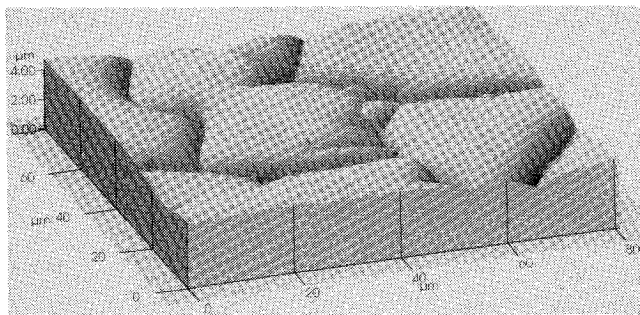


Fig. 13 Atomic-force-microscope topographic image of sample F2 after AO exposure on STS-46.

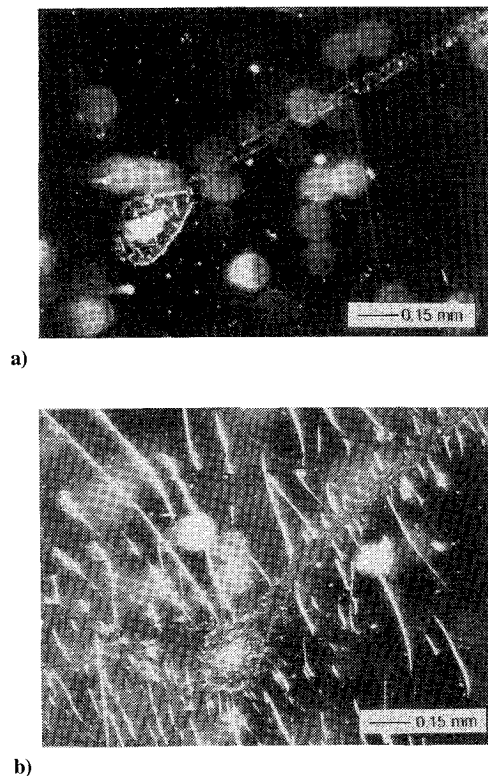
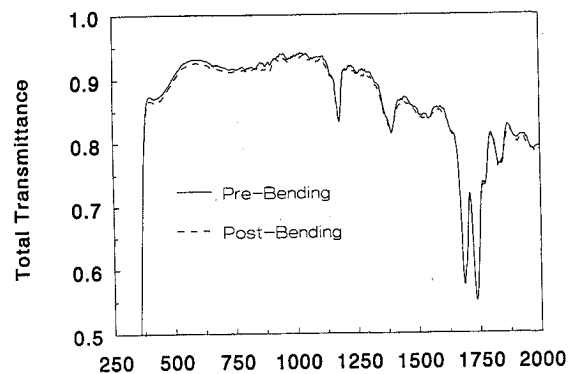


Fig. 14 Uncoated DC 93-500 exposed to LEO on STS-46: a) before flight (no cracks) and b) after flight (SiO_x conversion cracks).

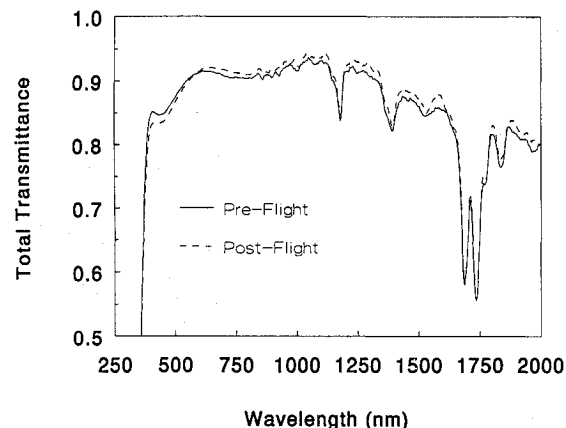
bending. The total transmittance spectrum of sample F2, however, shows a slight decrease between approximately 375 and 500 nm, and a slight increase between approximately 625 and 2125 nm (see Fig. 15). The specular transmittance spectra of the bend-test sample showed a fairly uniform drop across the range of 250–2125 nm. In contrast, the specular transmittance spectra of sample F2 showed a larger decrease in the lower wavelengths of this range (≈ 350 –700 nm) and a smaller decrease in the higher wavelengths (≈ 1300 –2125 nm). These spectral differences suggest that there is an additional mechanism affecting the optical properties of the AO-exposed samples besides the additional cracking. Because these changes do appear to occur in the spectra of sample G7 at the lowest fluence interval ($F = 5.2 \times 10^{20}$ atoms/cm²), it is possible that a contaminant film had started to develop on the flight sample. The transmittance data for sample F2 did not show an interference pattern, but the interference pattern was also not present in the spectra of sample G7 until a fluence of 1.0×10^{21} atoms/cm². The EOIM-III fluence, 2.3×10^{20} atoms/cm², would be equivalent to approximately 21 h of ground-laboratory exposure.

Discussion

Modified techniques have been found to significantly decrease the number of metal oxide coating cracks formed during coating deposition. This study provides evidence that coating cracks will



a) Before and severe bending



b) Pre- and postflight

Fig. 15 Comparison of total transmittance spectral changes of coated DC 93-500 exposed to severe bending and to LEO AO.

propagate at temperatures as low as 40°C. Silicone contamination from the exposed silicone substrate at coating crack sites poses a danger to the Fresnel lens and to surrounding spacecraft systems that depend on its optical properties. If means are not developed to prevent the formation of these cracks, then multilayer coated silicone does not appear to be promising for LEO applications with higher operating temperatures. However, more recent studies have shown that the operating temperature range of the minidome Fresnel lenses in LEO is more likely to be between -29°C and 10°C and is very dependent on the concentrator array design. In LEO application, in which the operating temperatures are between -29°C and 10°C , the multilayer coating may be acceptable if the pristine lenses have a minimal number of cracks. Low-temperature thermal exposures and thermal cycling have not been performed to evaluate the effects of these low temperatures on the integrity of the coating. Further testing should be done to study the effects of these conditions on the coated silicone lens material. Also, testing should be done to evaluate the effects that stresses place on the coating due to thermal gradients in the refractive concentrators as a potential source of coating cracks.

The changes observed in flight sample F2 were minor because of the relatively low AO fluence (2.3×10^{20} atoms/cm²) of the EOIM-III mission. This low fluence makes comparisons between in-space and ground-based AO exposures difficult. Longer-term space-flight exposures of refractive concentrator materials, and ground-laboratory and in-space exposure of actual refractive concentrators, are needed to verify the long-term effects of AO, ultraviolet radiation, and thermal cycling on the optical properties of refractive concentrators. Two passive minidome Fresnel lenses were recently flown on STS-60, and an active experiment, the Photovoltaic Array Space Power Plus Diagnostics Flight Experiment,¹ was also recently flown on the Advanced Photovoltaic and Elec-

tronics Experiment (APEX). Both of these space-flight experiments have multilayer metal-oxide-coated silicone concentrators.

Problems with excessive sample heating in an AO plasma asher can make distinguishing between AO effects and heating effects on samples that are sensitive to high temperature and/or temperature variations difficult. In order to accurately study the effects of AO on this type of coated silicone material, a method of cooling and stabilizing the temperature of a sample within a plasma asher must be devised in order to avoid cracking due to thermal stresses, which affects the optical properties of the samples and may be mistaken for or mask AO effects.

Conclusions

Multilayer metal-oxide-coated silicone refractive concentrator material was exposed to ground-laboratory and in-space UV-containing AO environments. Ground-laboratory exposures indicated a slight drop in the total transmittance initially (0.834 to 0.796), but it became stable at 0.793 with further exposure (decrease of 0.041). The specular transmittance was found to decrease continuously with exposure (0.749 to 0.606, a decrease of 0.143). Coating cracks present on the pristine sample were found to continue to develop with environmental exposure. Propagation of coating cracks was found to be caused by heating to temperatures as low as 40°C. Bend-test results show that decreases in total and specular transmittance up to 0.005 and 0.023, respectively, could be attributed to additional cracking. Interference fringe patterns in the spectral data indicated the development of a contamination layer during exposure. The source of contamination is most likely the reaction of AO with exposed silicone at coating crack sites (and other unprotected silicone) in a UV-containing environment. Mass measurements, SEM analysis, and XPS data support the formation of surface SiO_x at exposed silicone areas in coating crack sites. Evidence of silicone-to- SiO_x conversion in coating cracks, in addition to the knowledge of results such as those on LDEF, indicates that a by-product of silicone contamination is likely to have occurred, resulting in a contaminant film that contributed to total and specular transmittance loss.

As a result of the CTE mismatches between the metal oxide protective coating and silicone substrate, and the demonstrated potential for silicone contamination, alternative substrates with lower CTEs and that do not produce contaminant by-products should be considered for LEO applications.

Acknowledgments

The authors would like to thank Michael F. Piszczor of NASA Lewis for his support on this program, the Boeing Defense and Space Group for supplying the materials for both ground-laboratory and space-flight exposure, Judith A. Terlep of Ohio Aerospace Institute for preflight microscopy of the flight samples, and Jeff Shallenberger at Evans East for conducting the extensive XPS analyses.

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