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Evaluation of Space Power Materials Flown on the Passive Optical Sample Assembly

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Abstract

Evaluating the performance of materials on the exterior of spacecraft is of continuing interest, particularly in anticipation of those applications that will require a long duration in low Earth orbit. The Passive Optical Sample Assembly (POSA) experiment flown on the exterior of Mir as a risk mitigation experiment for the International Space Station was designed to better understand the interaction of materials with the low Earth orbit environment and to better understand the potential contamination threats that may be present in the vicinity of spacecraft. Deterioration in the optical performance of candidate space power materials due to the low Earth orbit environment, the contamination environment, or both, must be evaluated in order to propose measures to mitigate such deterioration. The thirty two samples of space power materials studied here include solar array blanket materials such as polyimide Kapton H and SiO_x coated polyimide Kapton H, front surface aluminized sapphire, solar dynamic concentrator materials such as silver on spin coated polyimide and aluminum on spin coated polyimide, CV1144 silicone, and the thermal control paint Z-93-P.

The physical and optical properties that were evaluated prior to and after the POSA flight include mass, total, diffuse, and specular reflectance, solar absorptance, and infrared emittance. Additional post flight evaluation included scanning electron microscopy to observe surface features caused by the low Earth orbit environment and the contamination environment, and variable angle spectroscopic ellipsometry to identify contaminant type and thickness. This paper summarizes the results of pre- and post-flight measurements, identifies the mechanisms responsible for optical properties deterioration, and suggests improvements for the durability of materials in future missions.

Introduction

Materials located on the exterior of spacecraft in low Earth orbit are subjected to a number of environmental threats, including atomic oxygen, ultraviolet radiation, thermal cycling, and meteoroid and debris impact[1-4]. Atomic oxygen attacks materials vulnerable to oxidation, particularly organic materials such as Kapton. Ultraviolet radiation can break chemical bonds and cause undesirable changes in optical properties. Repeated thermal cycling can cause cracking and spalling of coatings, and meteoroid and debris impacts can damage protective coatings. Another threat to materials on the exterior of spacecraft is contamination[5]. The outgassing of volatile chemicals can cause contamination of nearby surfaces, changing their optical and thermal control properties. Contaminated surfaces may undergo further change as a result of atomic oxygen and ultraviolet radiation exposure.

The Passive Optical Sample Assembly (POSA) experiment, part of the Mir Environmental Effects Payload (MEEP), was designed as a risk mitigation experiment for the International Space Station. By characterizing samples initially, exposing them for a long duration on the exterior of Mir, and characterizing samples upon their return, lessons learned about material properties durability can be applied to the International Space Station and other long duration missions. Enough samples were contributed by various investigators to warrant two carriers, POSA I and POSA II, with each carrier having accommodations for both Mir facing samples and space facing samples.

This paper will present the pre-flight and post-flight results from seven different sample types, with each type having a specific goal. Conclusions will be identified from the results obtained on each of the specific samples and general conclusions will be identified to benefit future long duration missions.

Background

The Mir Environmental Effects Payload (MEEP) was a set of four experiments flown on the Russian Space Station Mir, for a total of 18 months, from late March 1996 (deployed on STS-076) through late September, 1997 (retrieved on STS-086). The experiments included two meteoroid and debris impact experiments, and two passive materials exposure experiments.

The two materials exposure experiments were each part of an International Space Station contamination risk mitigation activity. The Passive Optical Sample Assembly (POSA) experiments were identified as POSA I and POSA II. NASA Marshall Space Flight Center was the technical lead for POSA I. This experiment contained over 200 test specimens on each of its two sides. No silicone containing specimens were permitted on this experiment. Boeing was the technical lead for POSA II and NASA Glenn Research Center was a participant. This experiment contained over 250 samples on each of its two sides. POSA II contained a number of silicone, fluorosilicone, and silicone-coated specimens, along with specimens of several aluminum anodizes and white paints.

Identical sets of material specimens were placed on opposite sides of each POSA experiment. The trays were mounted inside a closed ~2' by 2' by 6"container, much like a suitcase that was hinged on one side. The MEEP payloads were carried to Mir using the Space Shuttle, with the closed containers mounted in side wall carriers in the cargo bay. Each container was deployed on-orbit during an extra vehicular activity by two U.S. Astronauts. Each container had a shaft attached to the edge opposite the hinges so that the container could be clamped to a handrail on the docking module. Once the containers were attached to the docking module, they were opened such that the trays holding the samples were facing in opposite directions. POSA I was mounted on the nominal "leading edge" side of the docking module, with one side oriented about 45° from the ram direction, looking generally away from Mir. The other side of POSA I had a large view factor to many of the Mir modules. POSA II was mounted on the nominal "trailing edge" side of the docking module, with one side oriented toward Mir and the other side oriented with a view along the docking module to space. The attitude of Mir changed numerous times during the 18 month exposure and each side of both POSA I and POSA II were exposed to atomic oxygen at some point in the mission. Solar UV detectors were flown on each side of both POSA experiments. The UV detector on POSA II looking toward Mir failed during the mission. The other detectors indicated a solar UV exposure level of 413 equivalent sun hours for Mir facing POSA I, 571 equivalent sun hours for space facing POSA I, and 576 equivalent sun hours for space facing POSA II. The Mir altitude was about 300 km during the exposure period, at an orbital inclination of 51.6°.

Methods

Mass measurements on Kapton to determine atomic oxygen fluence were made on a Mettler microbalance, capable of measuring mass to the nearest 0.000001 g. Mass measurements for other samples were made on a Sartorius balance, capable of measuring mass to the nearest 0.00001 g. In all cases, samples were dehydrated under vacuum for at least 48 hours just prior to mass measurement.

Reflectivity measurements were made utilizing two instruments, a Perkin-Elmer Lambda-9 spectrophotometer, and an AZ Technology Laboratory Portable Spectro-Reflectometer (LPSR). Both instruments operate in the wavelength range of 250 to 2500 nm. Both instruments measure spectral reflectance, used to calculate solar absorptance. The Perkin-Elmer Lambda-9 spectrophotometer offers the added advantage

of providing specular and diffuse reflectance based on a 15 cm diameter Spectralon integrating sphere. Total reflectance at air mass zero was obtained by convoluting the total spectral reflectance with the air mass zero solar spectrum. Total reflectance at air mass zero was subtracted from unity to yield solar absorptance[6].

Infrared reflectance measurements were made in the vicinity of 9.7 μm utilizing a Gier-Dunkle DB-100 infrared reflectometer. For opaque samples, the infrared reflectance provided by the DB-100 is subtracted from unity to yield emittance. The sample remains at room temperature. Hence, the emittance provided is a room temperature emittance value[7].

Selected samples were subjected to Variable Angle Spectroscopic Ellipsometry (VASE) for the purpose of identifying the thickness of contaminants. VASE covering a spectral range of 0.2 to 14 microns was used to determine the chemical structure and thickness of the contaminant layer on one Mir facing sample, one space facing sample, and one control.

Other samples were subjected to scanning electron microscope (SEM) evaluation to obtain images of atomic oxygen erosion, etc.

Samples and Objectives

Polyimide Kapton H was selected for mass loss measurements, to calculate atomic oxygen fluence. SiO_x coated Kapton H solar array blanket material was selected for atomic oxygen undercutting studies. Front surface aluminized sapphire was selected to measure degradation optically as a result of contamination. Silver on spin-coated polyimide was selected to measure specular reflectance degradation as a result of atomic oxygen attack at defect sites. Aluminum on spin-coated polyimide was also selected to measure specular reflectance degradation as a result of atomic oxygen attack at defect sites. CV1144 silicone was selected to look for embrittlement as a result of UV exposure. Finally, Z-93-P thermal control paint was selected to evaluate emittance and absorptance changes. All samples were 2.54 cm in diameter. The edges of each sample were hidden by the chamfered sample holder hardware. Hence, the total surface area of each sample exposed to the space environment was approximately 3.40 cm^2. The quantity of each sample type is as follows. There were four Kapton H samples, two Mir facing and two space facing. There were four SiO_x coated Kapton H samples, two Mir facing and two space facing. There were six front surface aluminized sapphire samples, three Mir facing and three space facing. There were four silver on spin coated polyimide and four aluminum on spin coated polyimide samples, with each type having two Mir facing and two space facing samples. There were six CV1144 samples, three Mir facing and three space facing. Four out of six of the CV1144 samples were purposely covered with sacrificial Kapton. Finally, there were four Z-93-P samples, two Mir facing and two space facing samples, for a total of 32 flight samples. In addition, there was a ground control for each sample type.

Results and Discussion

General Observations:

Figure 1a shows the POSA II package being opened after flight. In general, the appearance of the returned samples was as expected. There was evidence of atomic oxygen erosion on the unprotected Kapton samples, and there was some discoloration on samples and mounting hardware. It was interesting to note that some regions were not discolored, apparently protected from the source of the discoloring agent due to shadowing. The degree of atomic oxygen erosion was obviously less than expected, because pieces of sacrificial Kapton remained in place. The most obvious and unexpected feature was found on the space facing side of the package, where as shown in Figure 1b there was a spattering across the surface best characterized as the dried remains of droplets. These dried remains are thought to have come from one or more waste water dumps from the space shuttle, while docked to Mir, hereafter referred to as the organic contaminant.

Figure 1a. POSA II package being opened after flight.

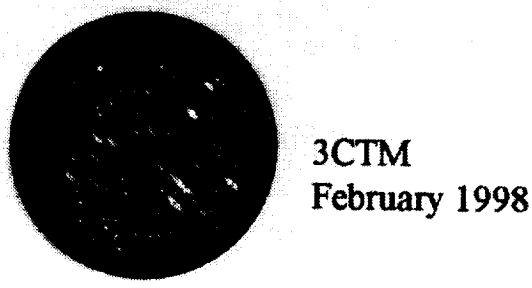

Figure 1b. Close up of one sample from the space facing side, showing the spattering of droplets.

Kapton:

Figure 2 summarizes the atomic oxygen fluence measured by the two Mir facing and the two space facing Kapton samples. The two Kapton samples on the Mir facing side of the POSA-2 package both had a mass loss near 0.0012 grams, yielding an estimated atomic oxygen fluence of 8.2×10^{19} atoms/cm^2. The two Kapton samples on the space facing side of the POSA II package both had a mass loss near 0.0031grams, yielding an estimated atomic oxygen fluence of 2.1×10^{20} atoms/cm^2. It is interesting to note that the SiO_x coated Kapton H showed no change in dehydrated mass. With the protective coating of SiO_x, the atomic oxygen fluence was found to be too small for undercutting studies. The absence of change in mass further suggests that the mass of contaminants found on these (and all the other samples including the Kapton samples) was negligibly small.

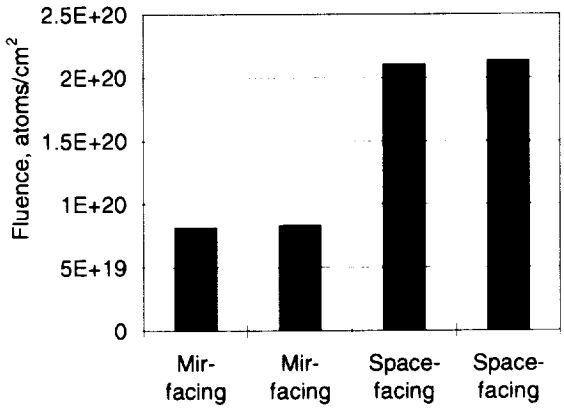

Figure 2. Atomic oxygen fluence measured by Kapton.

Front Surface Aluminized Sapphire:

Figure 3 shows that the total reflectance of the front surface aluminized sapphire samples decreased as a result of exposure on Mir. On the Mir facing side, the total reflectance decreased, on average, from 0.900 to 0.863. On the space facing side, the total reflectance decreased, on average, from 0.900 to 0.877. Both of these changes suggests a contaminant on the surface, with the thickness of the contaminant being greater on the Mir facing side compared to the space facing side. This non organic contaminant was identified and quantified by variable angle spectroscopic ellipsometry (VASE) at the University of Nebraska ñ Lincoln.

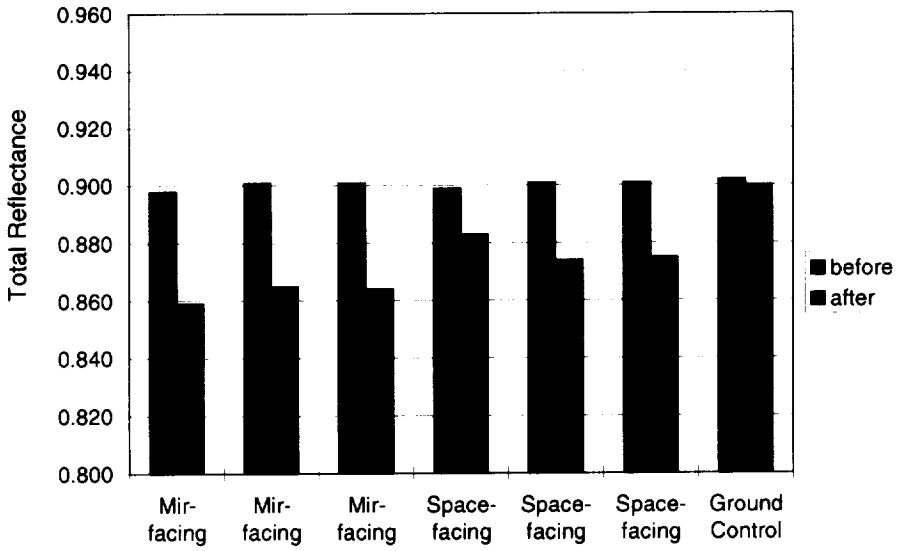

Figure 3. Total reflectance of front surface aluminized sapphire.

VASE is an optical characterization technique used for thin film measurements[8-10]. It is typically used to determine thin film thickness and refractive index (n and k). A unique feature of VASE in the infrared is the ability to obtain chemical bond information from very thin films. Chemical bonds absorb at select frequencies in the infrared; thus by mapping absorption bands to their corresponding chemical bonds one can determine the chemical structure of a material. Using VASE in the infrared, the imaginary part of the refractive index (k) of the contamination layer was measured, which is essentially the normalized absorption in a material. The molecular bond absorption bands present in the measured refractive index suggest that the contamination is SiO_2, hereafter referred to as the inorganic contaminant.

In Figure 4 the measured contaminant optical constants are compared to quartz optical constants from the literature[11], showing that the molecular bond absorptions match very well. Table 1 gives the measured contamination layer thickness for the Mir facing and space facing samples. The thickness given for the space facing sample is an average of the four locations measured. The larger thickness of the Mir facing sample suggests that this sample was more in the line of sight of the contamination source.

Figure 5 shows that the diffuse reflectance of the Mir facing samples remained essentially unchanged, at 0.011, while the space facing diffuse reflectance increased from 0.011 to a maximum of 0.053, undoubtedly due to the organic contaminant mentioned earlier.

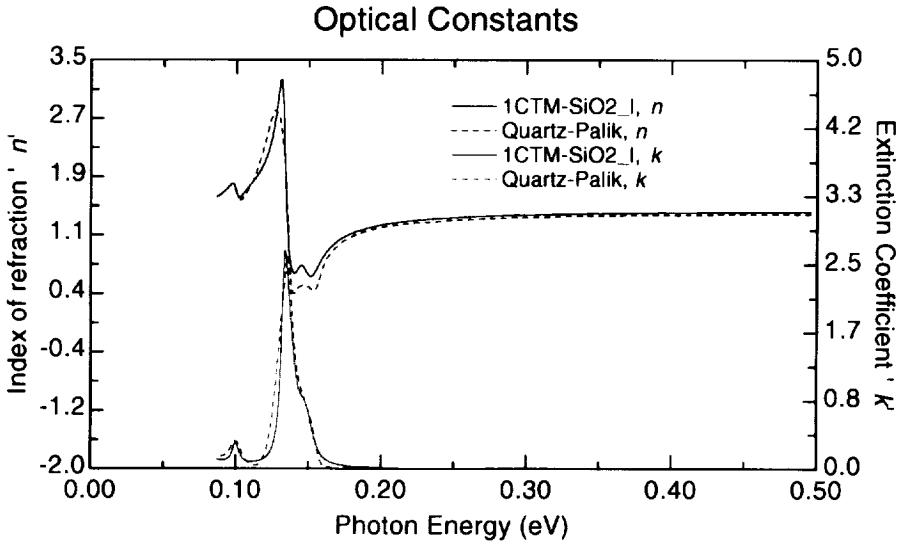

Figure 4. Comparison of measured contamination optical constants with quartz optical constants from the literature, suggesting SiO_2contamination.

Table 1. Contamination layer thickness for select post retrieval front surface aluminized sapphire samples.

Sample	Contamination Layer Thickness (Å)	90% Confidence Limits
Mir Facing	235.0	±0.5 Å
Space Facing	53.5	±10 Å

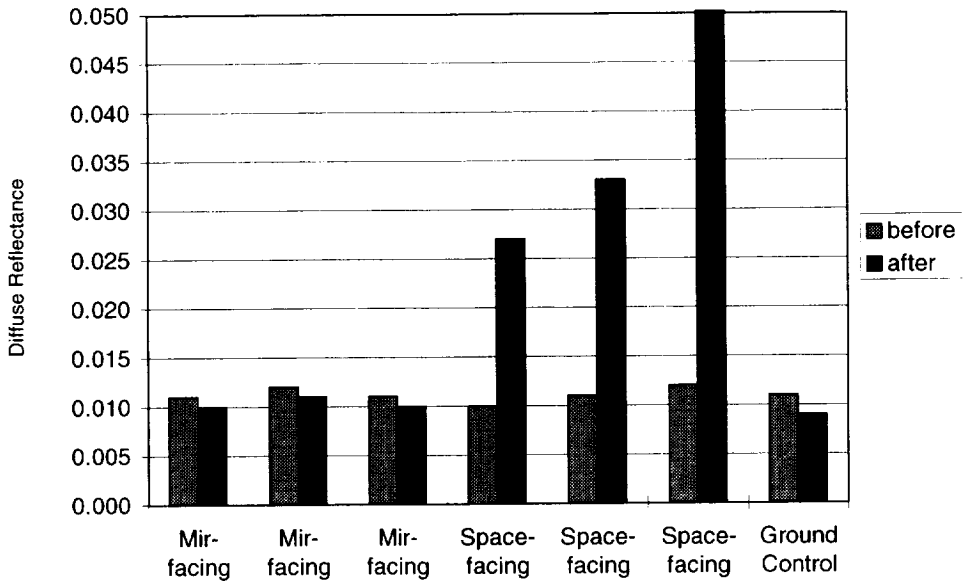

Figure 5. Diffuse reflectance of front surface aluminized sapphire.

As shown in Figure 6, the inorganic contaminant had the impact of increasing the solar absorptance of these samples, from 0.100 to 0.137 on the Mir facing side, and from 0.100 to 0.123 on the space facing side. The organic contaminant is thought to be responsible for the increase in emittance observed on the space facing samples, as seen in Figure 7.

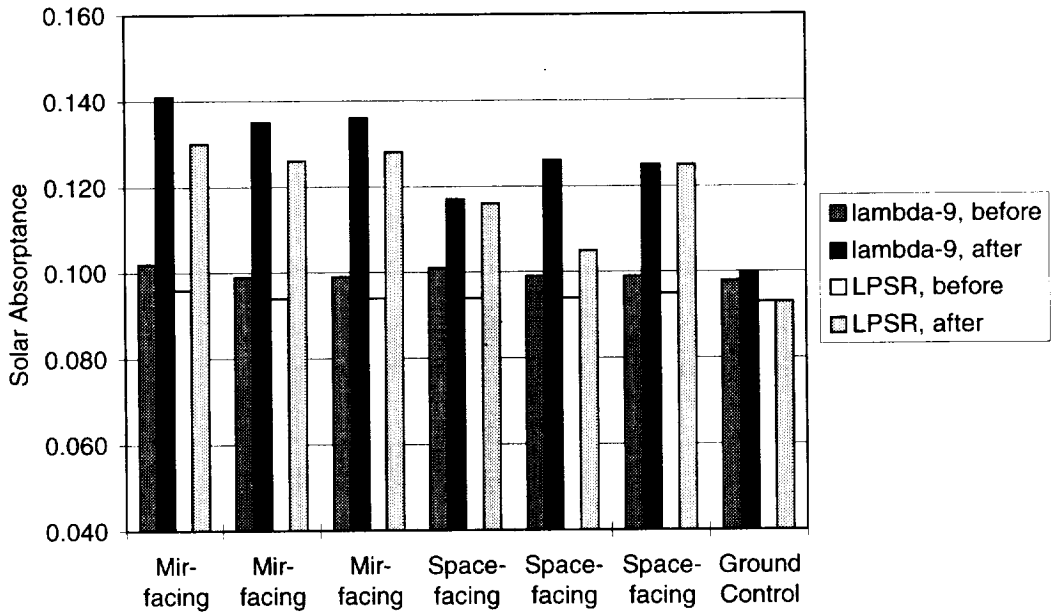

Figure 6. Solar absorptance of front surface aluminized sapphire.

Aluminum on Spin-Coated Polyimide:

Only minor changes in the total reflectance of the aluminum on spin-coated polyimide were observed, as shown in Figure 12. Unlike the silver, there was no appreciable oxidation of the aluminum in the ground control, at least as indicated by total reflectance. Figure 13 shows that some changes were observed in diffuse reflectance, with the space facing diffuse reflectance exhibiting a substantial increase due to the organic contaminant. There was no appreciable change in solar absorptance for any of the samples, as shown in Figure 14. Emittance increased slightly for the space facing samples, as shown in Figure 15.

The partially eroded Kapton was successfully used for scanning electron microscopy (SEM). Features from the Mir facing samples are summarized in Figure 16. The Kapton was both textured and covered with contaminant. One SEM photograph showed a particle on the surface and indicates the oblique angle of atomic oxygen arrival, where some material was protected from atomic oxygen on the shadowed side of the particle. One crater-like impact site was found. The textured/contaminated surface of the Kapton was interrupted by an occasional splatter pattern. Further texturing seems to have occurred in the center of the splatter patterns. Features from the space facing sample are summarized in Figure 17. In this case, the Kapton was noticeably textured. One region showed some streaking, similar to the splatter pattern seen on the Mir facing side. However, the streaking on the space facing side is better characterized as texture that was somehow knocked down. Another particle found on the surface, shows a protected "mesa" with atomic oxygen texturing in the background. An SEM photograph of the dried organic contaminant is shown. Also shown is a region where the organic contaminant was purposely washed off. It is interesting to note the step at the interface between the area of erosion and the area of Kapton previously covered by contaminant, suggesting that the contaminant was present for some time.

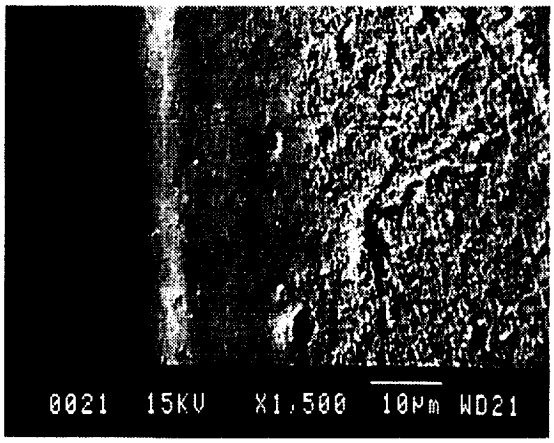

Figure 16a. Protected, shadowed, and unprotected Kapton, Mir facing.

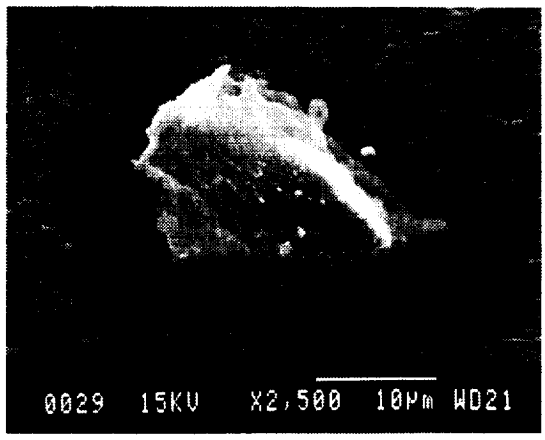

Figure 16b. Particle showing oblique angle of atomic oxygen arrival.

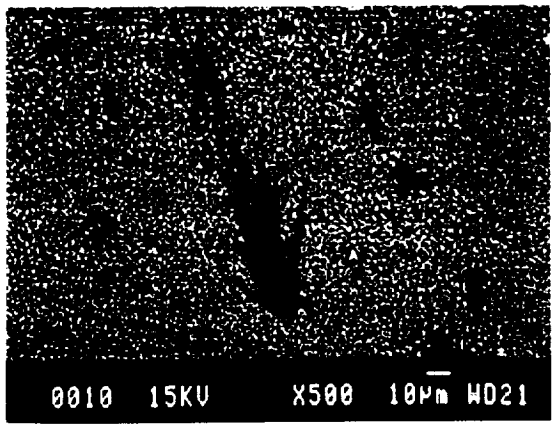

Figure 17a. Streaking of the atomic oxygen texturing.

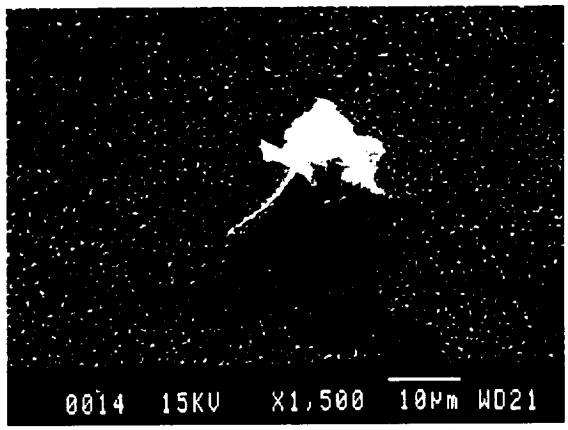

Figure 17b. Protected "mesa" with atomic oxygen texturing in the background.

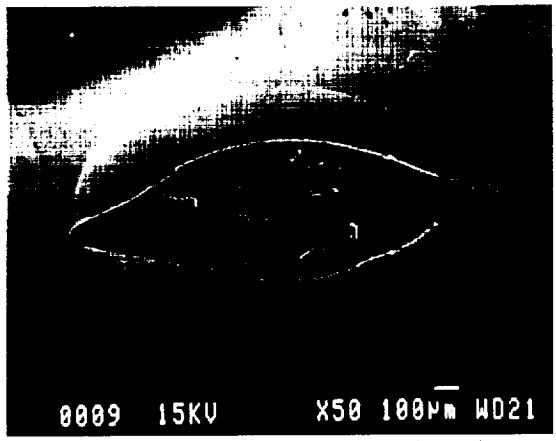

Figure 17c. Organic contaminant, uncleaned.

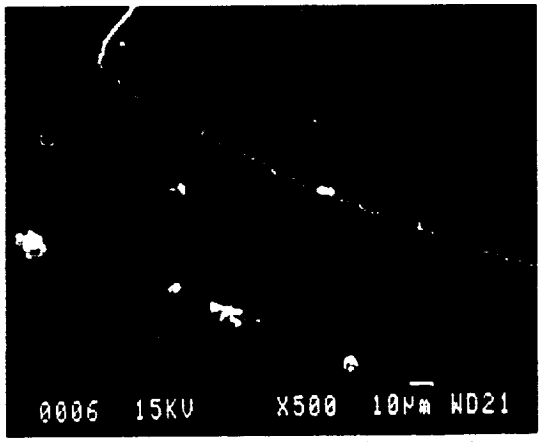

Figure 17d. Texturing near organic contaminant, after cleaning.

Z-93-P:

There was little to no change in the solar absorptance of the Z-93-P samples characterized by Glenn, and only a small change in their infrared emittance. Figure 18 summarizes the pre-flight vs. post-flight solar absorptance for several Z-93-P samples on POSA II, including the Glenn samples. Figure 19 summarizes the pre-flight vs. post-flight infrared emittance for the same samples. The decrease in infrared emittance was less pronounced in Glenn samples compared to the other samples on POSA II.

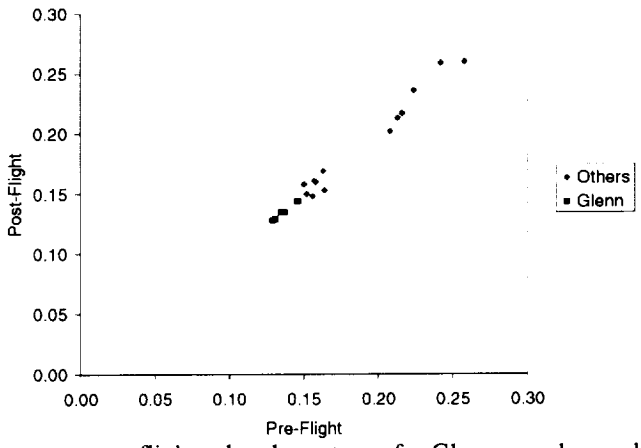

Figure 18. Pre-flight versus post-flight solar absorptance for Glenn samples, and others on POSA II.

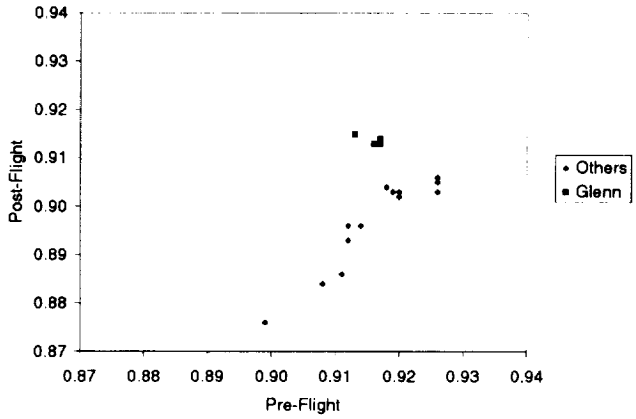

Figure 19. Pre-flight versus post-flight emittance for Glenn samples, and others on POSA II.

Discussion

A detailed discussion of the nature of the organic contaminant is beyond the scope of this paper. Briefly, the organic contaminant is thought to have come from a waste water dump containing urea. The organic contaminant was only found on the space facing samples, which happened to have a view of the shuttle when it was docked with Mir. The shuttle visited Mir three times while POSA was deployed. Even when docked, the shuttle typically performed a waste water dump every couple of days. One or more encounters with the waste water plume could be responsible for the organic contamination found on the space facing POSA samples.

The source for the inorganic contaminant is consistent with a silicone, released as an organic silicone vapor and ultimately oxidized by atomic oxygen to SiO_2. Background outgassing rates, as measured by other POSA investigators, suggest an outgassing rate in the vicinity of Mir on the order of 10^{-11} g/cm^2/sec, with increased outgassing rates during periods of zero eclipse orbits. Three such zero eclipse orbits occurred while POSA was deployed. During zero eclipse orbits, the exterior surfaces are illuminated throughout the entire orbit. Because no eclipse is encountered to allow the exterior surfaces to cool, exterior surfaces tend to overheat during such episodes. In this case, the temperature of a recently installed solar array carrier painted with KO-5191 organic silicone based paint is thought to have increased, causing the outgassing episodes that were responsible for the silicone contamination. Interestingly, POSA II was not in the line of sight of the solar array carrier. However, silicone contamination was found on POSA II. The amount of contamination on POSA II (235 Å on the Mir facing side and 53.5 Å on the space facing side) was admittedly less than that found on POSA I (approximately 7000Å) suggesting that the silicone vapor may have some mobility in non line of sight directions.

To mitigate risk on future long duration spacecraft in low Earth orbit, the obvious conclusions to draw from this work are to remove the sources of organic and inorganic contamination. Waste water dumps should be minimized and performed in such a way as to further reduce the chance for subsequent encounters. Paints with low outgassing performance should be utilized and components should be vacuum baked before or during assembly. Atomic oxygen on orbit may provide some cleaning of hydrocarbon contamination but should not be relied upon for removing all contaminants. More work is needed to understand the synergistic roles of atomic oxygen exposure, UV exposure, and other space environmental effects.

Conclusions

Space power materials on the exterior of spacecraft must be durable to both environmental threats and the threat of contamination. The passive optical sample assembly experiments flown on the exterior of Mir for 18 months revealed the impact of the space environment and the impact of contamination on the optical and physical properties of several materials. Atomic oxygen degradation was present, but was not as severe as expected. Of particular concern was the outgassing that occurred from silicone based paint during excessive heating on one or more zero eclipse orbits. Selecting a paint with better outgassing characteristics should reduce the impact of contamination caused by such overheating. Another concern was the release of waste water in the vicinity of Mir. Such waste water dumps must be carried out so that spacecraft surfaces do not encounter the plume. Additional work is needed to understand combined and synergistic environmental effects.

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13. ABSTRACT ***(Maximum 200 words)***

Evaluating the performance of materials on the exterior of spacecraft is of continuing interest, particularly in anticipation of those applications that will require a long duration in low Earth orbit. The Passive Optical Sample Assembly (POSA) experiment flown on the exterior of Mir as a risk mitigation experiment for the International Space Station was designed to better understand the interaction of materials with the low Earth orbit environment and to better understand the potential contamination threats that may be present in the vicinity of spacecraft. Deterioration in the optical performance of candidate space power materials due to the low Earth orbit environment, the contamination environment, or both, must be evaluated in order to propose measures to mitigate such deterioration. The thirty two samples of space power materials studied here include solar array blanket materials such as polyimide Kapton H and SiO_x coated polyimide Kapton H, front surface aluminized sapphire, solar dynamic concentrator materials such as silver on spin coated polyimide and aluminum on spin coated polyimide, CV1144 silicone, and the thermal control paint Z-93-P. The physical and optical properties that were evaluated prior to and after the POSA flight include mass, total, diffuse, and specular reflectance, solar absorptance, and infrared emittance. Additional post flight evaluation included scanning electron microscopy to observe surface features caused by the low Earth orbit environment and the contamination environment, and variable angle spectroscopic ellipsometry to identify contaminant type and thickness. This paper summarizes the results of pre- and post-flight measurements, identifies the mechanisms responsible for optical properties deterioration, and suggests improvements for the durability of materials in future missions.

14. SUBJECT TERMS

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