Analysis of Fluorinated Polyimides Flown on the Materials International Space Station Experiment

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March 2015
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Acknowledgments

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<th>Symbol</th>
<th>Description</th>
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<tr>
<td>AO</td>
<td>atomic oxygen</td>
</tr>
<tr>
<td>CP</td>
<td>colorless polyimide</td>
</tr>
<tr>
<td>ESH</td>
<td>equivalent Sun hour(s)</td>
</tr>
<tr>
<td>EXPRESS</td>
<td>EXpedite the PRocessing of Experiments to Space Station</td>
</tr>
<tr>
<td>GRC</td>
<td>Glenn Research Center</td>
</tr>
<tr>
<td>HPGT</td>
<td>high-pressure gas tank</td>
</tr>
<tr>
<td>ISS</td>
<td>International Space Station</td>
</tr>
<tr>
<td>LaRC</td>
<td>Langley Research Center</td>
</tr>
<tr>
<td>MgF$_2$</td>
<td>magnesium fluoride</td>
</tr>
<tr>
<td>MISSE</td>
<td>Materials on International Space Station Experiment</td>
</tr>
<tr>
<td>O</td>
<td>oxygen</td>
</tr>
<tr>
<td>PEC</td>
<td>passive experiment container</td>
</tr>
<tr>
<td>SiO$_x$</td>
<td>silicon oxide</td>
</tr>
<tr>
<td>UV</td>
<td>ultraviolet</td>
</tr>
<tr>
<td>VDA</td>
<td>vapor-deposited aluminum</td>
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1. INTRODUCTION

Since August 2001, the Materials on International Space Station Experiment (MISSE) have provided a wealth of space environmental effects data on a variety of materials and spacecraft components. Among these were samples of polymers developed at the Langley Research Center (LaRC), colorless polyimide 1 (CP1) and CP2. NeXolve Corporation, formerly known as SRS Technologies and ManTech Corporation, exclusively licensed the original polyimides from LaRC. Over the years, NeXolve modified CP1 by various proprietary additives and coatings to create new materials and improve film properties. This Technical Memorandum includes data on postflight structural integrity, visual observations, determination of atomic oxygen (AO) erosion yield (also known as AO reactivity, given in cm$^3$/atom), and optical property changes for samples returned to NASA Marshall Space Flight Center. More data may be found in reference 1 for polymeric materials studied at Glenn Research Center (GRC). Published MISSE data, photographs, and some raw data files are being gathered in a database in the Materials and Processes Technical Information System.

Unless otherwise specified, solar absorptance ($\alpha_s$) measurements were made with an AZ Technology Laboratory portable spectrophotometer model 300. Infrared emittance ($\varepsilon$) measurements were made with an AZ Technology TEMP 2000A infrared reflectometer. Changes of ±0.01 in these optical properties are not considered statistically significant. Transmission measurements were made with a Perkin Elmer Lambda 19 reflectometer prior to July 2012 and with a Perkin Elmer Lambda 1050 reflectometer after July 2012. Both reflectometers were equipped with 150-mm integrating spheres and provided comparable transmission measurements.
2. ENVIRONMENTAL DEFINITIONS

MISSE is a series of materials flight experiments, the first two of which were delivered to the International Space Station (ISS) during Space Transportation System-105 in 2001. Experiments developed by principal investigators were loaded onto hinged, suitcase-like containers, called passive experiment containers (PECs), and were exposed to the space environment on the exterior of the ISS. During transport to the ISS on the space shuttle, the PECs were closed with the samples facing each other for protection. Once the space shuttle reached the ISS, the PECs were attached to its exterior during an extravehicular activity and opened back to back, exposing the samples to space. Materials in this study were flown on MISSEs -1, -2, -3, -4, -6, and -7, which had various external locations on the ISS.

Table 1 presents their environments in terms of AO fluence (given as oxygen (O) atoms/cm²) and ultraviolet (UV) radiation in equivalent Sun hours (ESH). Figure 1 shows the ISS locations of these MISSEs. Note that the earlier MISSE flights were numbered for each PEC, so that MISSEs -1 and -2 flew together, as did MISSEs -3 and -4. The later flights were single numbers with different letters for each PEC, e.g., MISSEs -6A and -6B. No samples from MISSEs -5, -7A, or -8 are included in this study, though a study of other fluorinated polymers including Tedlar®, Tefzel®, Teflon®, and polyvinylidene difluoride from MISSE-5 may be found in reference 5.

Table 1. Mission exposure summary of MISSEs -1 through -7.

<table>
<thead>
<tr>
<th>MISSE</th>
<th>Placed Outside ISS</th>
<th>Retrieved From ISS</th>
<th>Exposure (yr)</th>
<th>Location on ISS</th>
<th>AO Fluence (O atoms/cm²)</th>
<th>UV Dose (ESH)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>8/16/01</td>
<td>7/30/05</td>
<td>3.95</td>
<td>HPGT</td>
<td>Wake: 1.1–1.3 x 10²⁰</td>
<td>4,500–5,600</td>
</tr>
<tr>
<td>2</td>
<td>8/16/01</td>
<td>7/30/05</td>
<td>3.95</td>
<td>Quest airlock</td>
<td>Ram: MgF₂ window block</td>
<td>Ram: 5,000–6,700</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Wake: 1.7–2 x 10²⁰</td>
<td>Ram: 5,000–6,700</td>
<td>Ram: 4,800–6,200</td>
</tr>
<tr>
<td>3</td>
<td>8/3/06*</td>
<td>8/18/07</td>
<td>1.04</td>
<td>HPGT</td>
<td>Wake: 1.9 x 10²⁰</td>
<td>Wake: 790</td>
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<tr>
<td>4</td>
<td>8/3/06*</td>
<td>8/18/07</td>
<td>1.04</td>
<td>Quest airlock</td>
<td>Ram: 2.1 x 10²¹</td>
<td>Ram: 1,590</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Wake: 3.6 x 10²⁰</td>
<td>Ram: 2,600</td>
<td>Wake: 995</td>
</tr>
<tr>
<td>6A, 6B</td>
<td>3/22/08</td>
<td>9/1/09</td>
<td>1.45</td>
<td>Columbus laboratory</td>
<td>Ram: 2 x 10²¹</td>
<td>Ram: 2,600</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Wake: 1.2 x 10²⁰</td>
<td>Ram: 2,400</td>
<td>Wake: 1,950</td>
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<tr>
<td>7B</td>
<td>11/23/09</td>
<td>5/20/11</td>
<td>1.49</td>
<td>EXPRESS Logistics Carrier 2 on the S3 truss</td>
<td>Ram: 4.2 x 10²¹</td>
<td>Ram: 2,400</td>
</tr>
</tbody>
</table>

*Deployed during Expedition 13.
All of the MISSE PECs for this study were flown in a ram/wake orientation, i.e., leading edge/trailing edge facing. These orientations provide different space environmental exposures. For example, samples in a ram orientation receive the greatest amount of AO exposure combined with solar radiation exposure, while those in a wake orientation receive solar radiation exposure with less AO exposure. The ISS has many orientations, including one for shuttle docking during which the MISSE wake side faced the ram direction. In general, the wake side samples received an order of magnitude less AO than the ram side samples. This means that, while material darkening related to UV radiation is sometimes more obvious on the wake side samples, these samples still experience AO erosion or bleaching. The exceptions to this are some thin film samples flown underneath a magnesium fluoride (MgF$_2$) window, which allowed 90% or more UV transmission while blocking any AO effects.

MISSE samples were exposed just past the solar cycle maximum and through the solar minimum. Thus, samples exposed on MISSE-7 were exposed to twice as much AO as the samples on MISSE-6, even though those on MISSE-6 were exposed for 1 month less than MISSE-7.

The reader should not assume that, because one material survived for a year on orbit, it would survive a year in any orbit, any environment, any orientation, or at any point in the solar cycle. It is critical to model the expected use environment and choose materials accordingly.

While severe molecular contamination can skew the results of a space environmental effects investigation, analysis of several optical witness samples flown on each MISSE indicated 50–500 Å of silicate deposition. This amount is enough to affect sensitive optics but not enough to significantly impact polymer erosion or optical properties.
3. COLORLESS POLYIMIDE 1

Three different versions of CP1 have been flown on MISSE—uncoated, coated on one side with vapor-deposited aluminum (VDA), and coated on one side with VDA and the other side with silicon oxide (SiO$_x$) (secs. 3.1–3.3, respectively).

3.1 Uncoated Colorless Polyimide 1

Uncoated CP1 was flown on the wake side of MISSEs -1 through -3. Figure 2 is the MISSE-1 sample, which is 1 mil thick with an adhesive-free solvent seam lap joint, which survived the $1.1 \times 10^{20}$ atoms/cm$^2$ AO fluence. A Kapton® witness sample was flown underneath the CP1 and indicated no through erosion. Figure 3 is the transmission measurements of the flight and control samples, which indicated no significant change due to exposure to the space environment. Measurements were made on either side of the lap joint.

Figure 2. MISSE-1 uncoated CP1 sample with solvent seam lap joint.
Figure 3. Transmission of MISSE-1 uncoated CP1.

Figure 4 is the MISSE-2 sample, which is 1 mil thick with an adhesive-free butt joint seam. While the sample survived the $\sim 2 \times 10^{20}$ atoms/cm$^2$ AO fluence, the seam failed after $\sim 1.8 \times 10^{20}$ atoms/cm$^2$, as indicated by the Kapton witness sample underneath. Measurements on either side of the butt joint showed an increase in transmission in the UV-visible wavelengths, confirming thinning of the material due to the higher AO fluence (fig. 5).

Figure 4. MISSE-2 uncoated CP1 with butt joint seam, with witness Kapton.
3.2 Colorless Polyimide 1 With Vapor-Deposited Aluminum

Aluminized CP1 was flown on MISSE-2. On the ram side, two identical 1-mil samples were flown underneath MgF$_2$ windows, and on the wake side, one 1-mil sample was directly exposed to space. These were all flown with the aluminized side exposed, which darkened due to UV exposure (fig. 6). Two samples of aluminized CP1 were also flown on the wake side of MISSE-3, one with the aluminized side exposed and the other with the CP1 side exposed. Figure 7 shows the reflectance curves for the samples where the aluminized side was exposed, flown on MISSE-2 (4-year exposure) and MISSE-3 (1-year exposure). The MISSE-3 wake sample with the CP1 exposed is shown in figure 8. Erosion was evident, as the infrared emittance dropped from 0.65 to 0.61. Figure 9 is the reflectance curve for the exposed polymer.
Figure 6. Aluminized CP1 with aluminized side exposed.

Figure 7. Reflectance of aluminized CP1 flown on MISSE-2 (4-year exposure) and MISSE-3 (1-year exposure). VDA was measured as first surface.
Figure 8. Aluminized CP1 flown with polymer side exposed.

Figure 9. Reflectance of aluminized CP1 with polymer side exposed. Reflectance is of VDA as second surface, through CP1.
In 2005, a solar sail of 2-µm CP1 with VDA was successfully tested at GRC. In addition to launch vibration and ascent vent tests, the 20-m² sail was deployed under high vacuum and 16 °C temperature in the Plum Brook Station test chamber. These tests led to the successful Nanosail-D2 mission in 2011. MISSE flight samples were made from the reserves of the 2-µm CP1 with VDA and flown behind windows on both the ram and wake sides of MISSE-6B. Some UV effects on reflectance can be seen in figure 10.

![Graph](image)

Figure 10. Reflectance of 2-µm CP1 with VDA.
3.3 Colorless Polyimide 1 With Vapor-Deposited Aluminum and Silicon Oxide

One of the MISSE-1 wake side samples was 5-µm-thick CP1 with VDA on one side, SiO$_x$ on the other side, and embedded Kevlar ripstop yarn. The SiO$_x$ side was exposed to space and $1.3 \times 10^{20}$ atoms/cm$^2$ of AO. This sample, including the Kevlar ripstop, survived the flight (fig. 11). Figure 12 is the reflectance curve for this coated CP1 sample.

Figure 11. MISSE-1 CP1 with VDA and SiO$_x$ coatings and Kevlar ripstop.
Figure 12. Reflectance of MISSE-1 CP1 with VDA, SiO$_x$, and ripstop.
A thicker sample without the ripstop was flown on the ram-facing side of MISSE-4 and exposed to $2.1 \times 10^{21}$ atoms/cm$^2$ of AO. The heavy AO attack seen in figure 13 resulted in some erosion through the 0.85-mil-thick film. The sample was flown with the SiO$_x$ coating exposed to space.

Figure 13. MISSE-4 CP1 with VDA and SiO$_x$ coatings.
4. CONDUCTIVE WHITE COLORLESS POLYIMIDE 1

Conductive white CP1 of 1.1 to 1.2 mil thickness was flown on the ram side of both MISSEs -6A and -6B, with the MISSE-6B sample flown under an MgF$_2$ window. This material is similar, but not identical, to the conductive material Thermalbright®. Atomic oxygen bleaching was noted (fig. 14), with a solar absorptance decrease from 0.442 to 0.371 due to $2 \times 10^{21}$ oxygen atoms/cm$^2$. Mass loss for this sample indicates an AO erosion yield of $9 \times 10^{-26}$ cm$^3$/atom. The sample flown under a window showed some UV darkening with an increase in solar absorptance from 0.448 to 0.532 (fig. 15).

Figure 14. MISSE-6A conductive white CP1.
Figure 15. Reflectance of MISSE-6 conductive white CP1.
5. CONDUCTIVE BLACK COLORLESS POLYIMIDE 1

Conductive black CP1 was flown on the ram side of MISSE-6A and did not survive the flight. It is likely that the black pigment was not adequately encapsulated to withstand AO erosion of the entire 1-mil thickness.
6. INFRARED TRANSPARENT COLORLESS POLYIMIDE 1

Two samples of 1.5-mil-thick infrared-transparent CP1 were flown on the wake side of MISSE-6B under two different window materials. The window for sample No. 1 transmitted more UV than the sample for No. 2, but no significant difference was noted between the samples. Figure 16 is the transmission curve for visible to near-infrared wavelengths for these samples. Figure 17 is the calculated infrared transmission for the 2- to 20-µm-wavelength band using an AZ Technology Laboratory portable infrared reflectometer. Infrared transmission was calculated using multiple infrared reflectance measurements made using high and low emittance materials as backing for the CP1 samples.

![Figure 16. Visible to near-infrared transmission of infrared transparent CP1.](image_url)
Figure 17. Calculated infrared transmission for infrared transparent CP1.
7. ESSAR STRETCH™ 255/EP2550 FILM

EP2550 film, now called Essar Stretch™ 255, was developed as a low modulus analog of CP1, capable of withstanding high temperatures, visibly clear, and easily modified with various fillers. Conductive (3.2 mil) and nonconductive (3.4 mil) versions were exposed directly to the space environment on the MISSE-6A ram side; nonconductive EP2550 samples were flown beneath windows on both ram and wake sides of MISSE-6B. The wake side sample was damaged during postflight handling.

Transmission of the nonconductive EP2550 is shown in figure 18. The directly exposed sample yellowed due to AO and UV interaction and had an AO erosion yield of $6.5 \times 10^{-26}$ cm$^3$/atom. The conductive version also darkened from a solar absorptance of 0.419 to 0.567 but had a lower AO erosion yield of $2.6 \times 10^{-26}$ cm$^3$/atom. Figure 19 shows the reflectance curve for the conductive EP2550.

![Figure 18. Transmission of MISSE-6 EP2550 samples.](image)
Figure 19. Reflectance of MISSE-6A conductive EP2550.
8. TOUGHENED COLORLESS POLYIMIDE 1

Toughened CP1 has improved tear resistance, as much as 40 times more resistant to tears than the original CP1. 0.28-mil-thick toughened CP1 was flown on both the ram and wake sides of MISSE-7B, but the ram side sample did not survive the flight. Figure 20 shows toughened CP1 and figure 21 shows the transmission of toughened CP1 samples flown on the wake side.

Figure 20. Toughened CP1 flown on MISSE-7B wake side.
Figure 21. Transmission of toughened CP1.
9. CORIN® XLS

An early version of CORIN® XLS labeled as an AO-resistant CP1 was flown on the MISSE-6A ram side. The AO erosion yield for this sample was $8.4 \times 10^{-26} \text{ cm}^3/\text{atom}$; the transmission is shown in figure 22.

Figure 22. Transmission of AO-resistant CP1.
0.26-mil-thick toughened CORIN® XLS was flown on both the ram and wake sides of MISSE-7B. The ram side sample was damaged but not entirely eroded away. The CORIN® XLS darkened more than the toughened CP1 flown in the same fixture (fig. 23). The transmission of toughened CORIN® XLS flown on the wake side is shown in figure 24.

Figure 23. Toughened CORIN® XLS flown on MISSE-7B wake side.

Figure 24. Transmission of toughened CORIN® XLS.
10. DISCUSSION AND CONCLUSIONS

The information presented here was gathered to support solar sail and advanced propulsion projects that might use colorless polyimide films. The specific exposure conditions and associated effects vary for each spacecraft/mission profile and must be considered when choosing materials, i.e., what works for low-Earth orbit conditions with minimal radiation effects may not work in a geosynchronous, Lagrangian, or a lunar environment. Some margin should be included in a design for unexpected degradation or contamination events. The MISSE experiments had low or localized contamination that did not significantly affect the results of space environmental effects. Proper materials selection and sensible contamination control procedures during hardware development, integration, and launch site processing should be enforced to minimize the impact of contamination on spacecraft performance.
REFERENCES


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**Abstract**

This Technical Memorandum documents the results from the Materials on International Space Station Experiment (MISSE) series involving fluorinated polyimide films analyzed at NASA Marshall Space Flight Center. These films may be used in thermal control, sunshield, solar sail, solar concentrator, and other lightweight polymer film applications. Results include postflight structural integrity, visual observations, determination of atomic oxygen erosion yield, and optical property changes as compared to preflight values.

### Subject Terms

polymer, atomic oxygen, ultraviolet radiation, solar sail, materials, lightweight

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