NASA/TM—2015–218205

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

M.M. Finckenor
Marshall Space Flight Center, Huntsville, Alabama

L. Rodman and B. Farmer
NeXolve Corporation, Huntsville, Alabama

March 2015

The NASA STI Program…in Profile

NASA/TM—2015–218205

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

M.M. Finckenor
Marshall Space Flight Center, Huntsville, Alabama

L. Rodman and B. Farmer
NeXolve Corporation, Huntsville, Alabama

National Aeronautics and
Space Administration

Marshall Space Flight Center • Huntsville, Alabama 35812

March 2015

Acknowledgments

The authors would like to acknowledge the hard work and dedication of the many scientists and engineers who have made the Materials International Space Station Experiment program so successful, especially Bill Kinard, Gary Pippin, Michael Stropki, Karen Gibson, Rob Walters, and Phil Jenkins. The authors also wish to thank Dr. Julie Robinson, Kevin Window, Rod Jones, Annette Sledd, and Ginger Flores for their support of International Space Station research. Thanks to Sandie Gibbs and Emmett Given for their talented photography, and to Don Burch and Curtis Bahr for laboratory support.

Available from:

NASA STI Information Desk
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681–2199, USA
757–864–9658

This report is also available in electronic form at
<http://www.sti.nasa.gov>

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF TABLES

LIST OF SYMBOLS, ACRONYMS, AND ABBREVIATIONS

AO	atomic oxygen
CP	colorless polyimide
ESH	equivalent Sun hour(s)
EXPRESS	EXpedite the PRocessing of Experiments to Space Station
GRC	Glenn Research Center
HPGT	high-pressure gas tank
ISS	International Space Station
LaRC	Langley Research Center
MgF_2	magnesium fluoride
MISSE	Materials on International Space Station Experiment
O	oxygen
PEC	passive experiment container
SiO_x	silicon oxide
UV	ultraviolet
VDA	vapor-deposited aluminum

TECHNICAL MEMORANDUM

ANALYSIS OF FLUORINATED POLYIMIDES FLOWN ON THE MATERIALS INTERNATIONAL SPACE STATION EXPERIMENT

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.[2]

Unless otherwise specified, solar absorptance (α_s) measurements were made with an AZ Technology Laboratory portable spectroreflectometer model 300. Infrared emittance (ε) 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/cm^2) and ultraviolet (UV) radiation in equivalent Sun hours (ESH).[3,4] 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.

MISSE	Placed Outside ISS	Retrieved From ISS	Exposure (yr)	Location on ISS	AO Fluence (O atoms/cm^2)	UV Dose (ESH)
1	8/16/01	7/30/05	3.95	HPGT	Wake: 1.1–1.3 x 10^{20}	4,500–5,600
2	8/16/01	7/30/05	3.95	Quest airlock	Ram: MgF_2 window block Wake: 1.7–2 x 10^{20}	Ram: 5,000–6,700 Wake: 4,800–6,200
3	8/3/06*	8/18/07	1.04	HPGT	Wake: 1.9 x 10^{20}	Wake: 790
4	8/3/06*	8/18/07	1.04	Quest airlock	Ram: 2.1 x 10^{21} Wake: 3.6 x 10^{20}	Ram: 1,590 Wake: 995
6A, 6B	3/22/08	9/1/09	1.45	Columbus laboratory	Ram: 2 x 10^{21} Wake: 1.2 x 10^{20}	Ram: 2,600 Wake: 1,950
7B	11/23/09	5/20/11	1.49	EXPRESS Logistics Carrier 2 on the S3 truss	Ram: 4.2 x 10^{21} Wake: 2.9 x 10^{20}	Ram: 2,400 Wake: 2,000

*Deployed during Expedition 13.

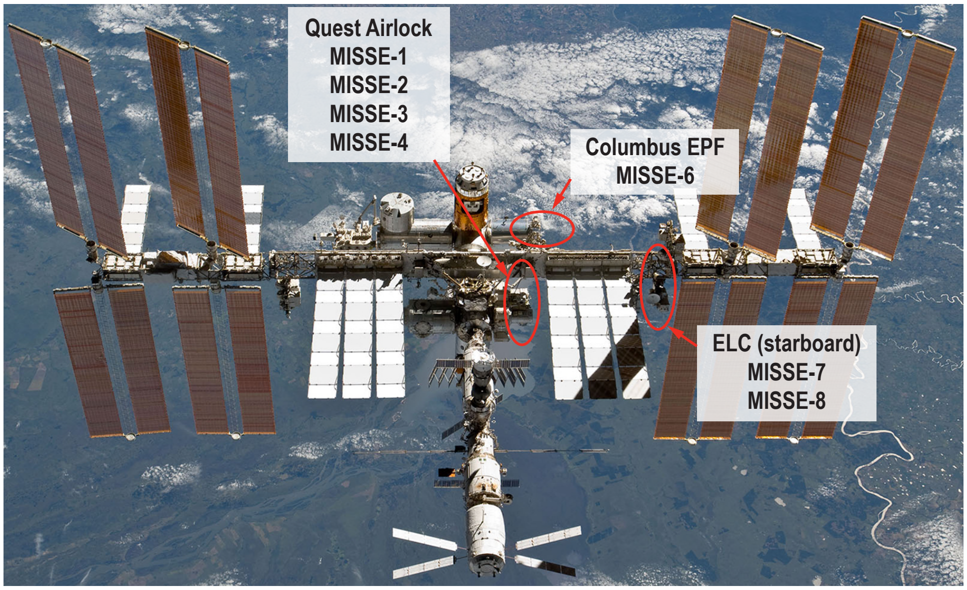

Figure 1. ISS locations of MISSEs -1 through -4 and -6 through -8.

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 $1.1-1.3\times10^{20}$ 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.

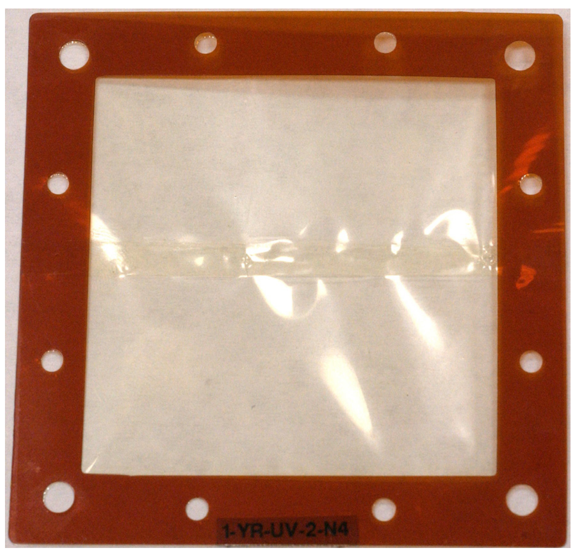

Figure 2. MISSE-1 uncoated CP1 sample with solvent seam lap joint.

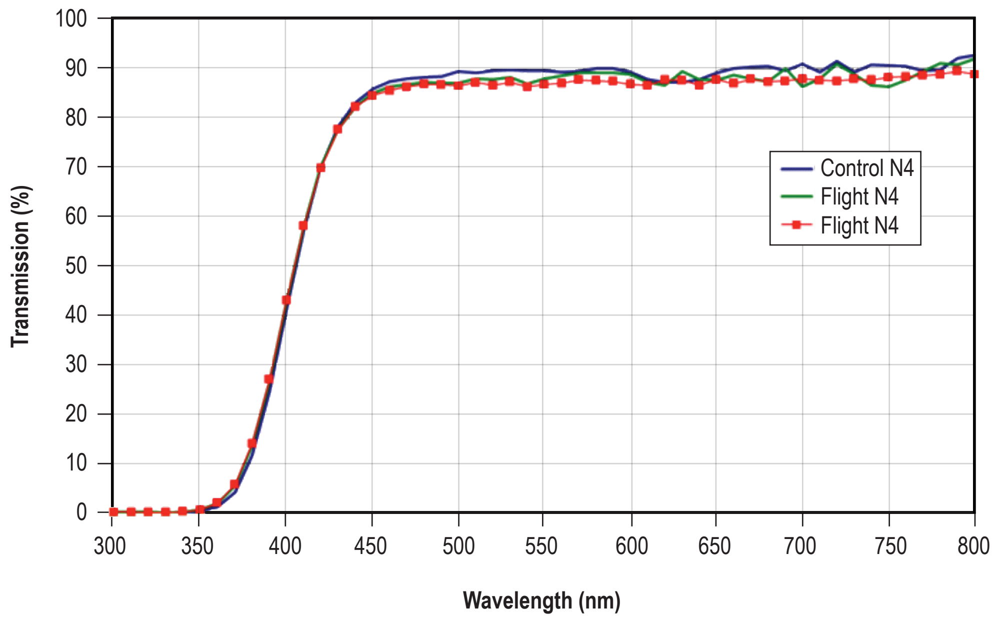

Figure 3. Transmission of MISSE-1 uncoated CP1.

Figure 4 is the MISSE-2 sample, which is 1 mil thick with an adhesive-free butt joint seam. While the sample survived the ~2×10^{20} atoms/cm^2 AO fluence, the seam failed after ~1.8×10^{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).

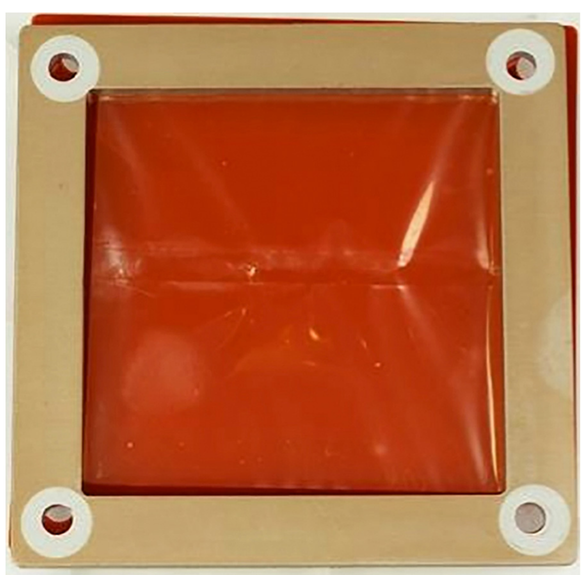

Figure 4. MISSE-2 uncoated CP1 with butt joint seam, with witness Kapton.

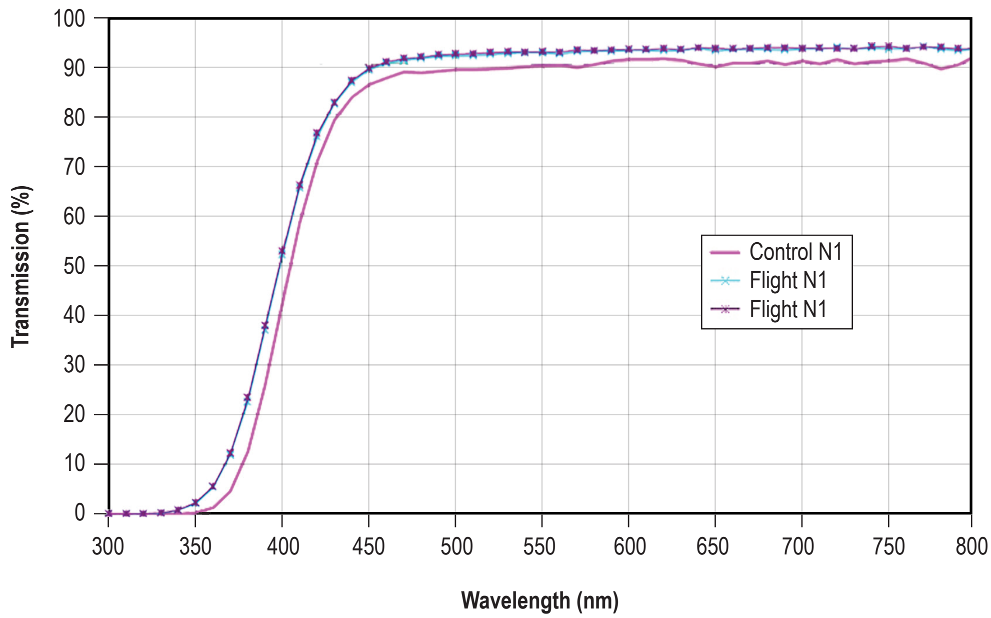

Figure 5. Transmission of MISSE-2 uncoated CP1.

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.

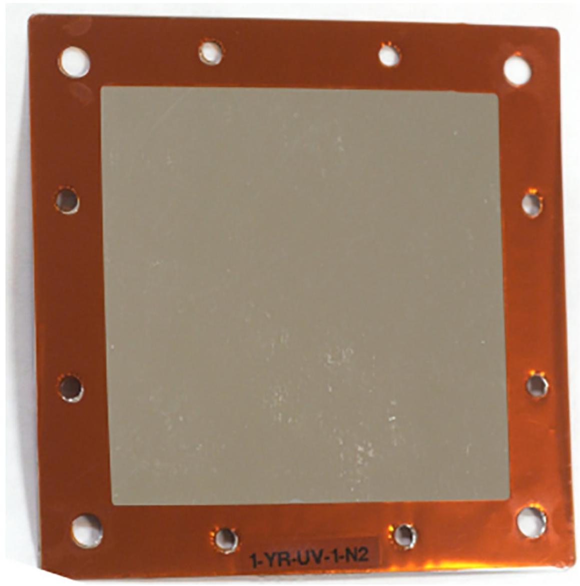

Figure 6. Aluminized CP1 with aluminized side exposed.

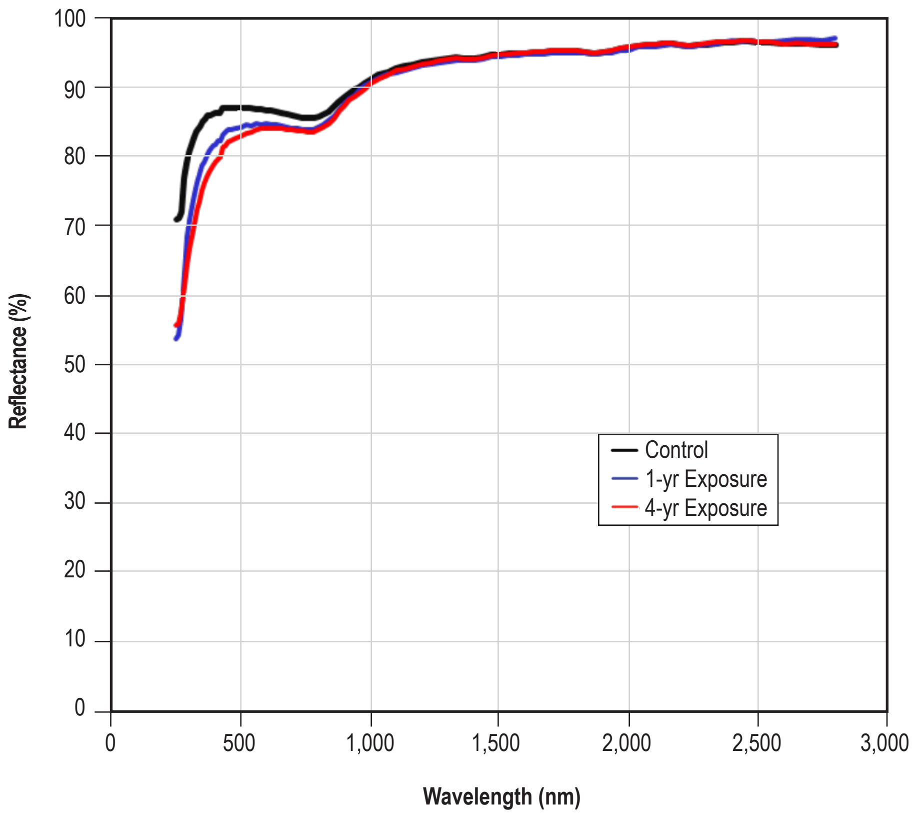

Figure 7. Reflectance of aluminized CP1 flown on MISSE-2 (4-year exposure) and MISSE-3 (1-year exposure). VDA was measured as first surface.

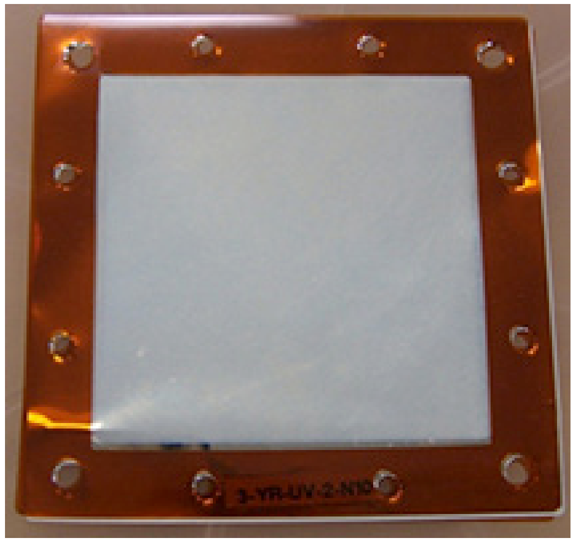

Figure 8. Aluminized CP1 flown with polymer side exposed.

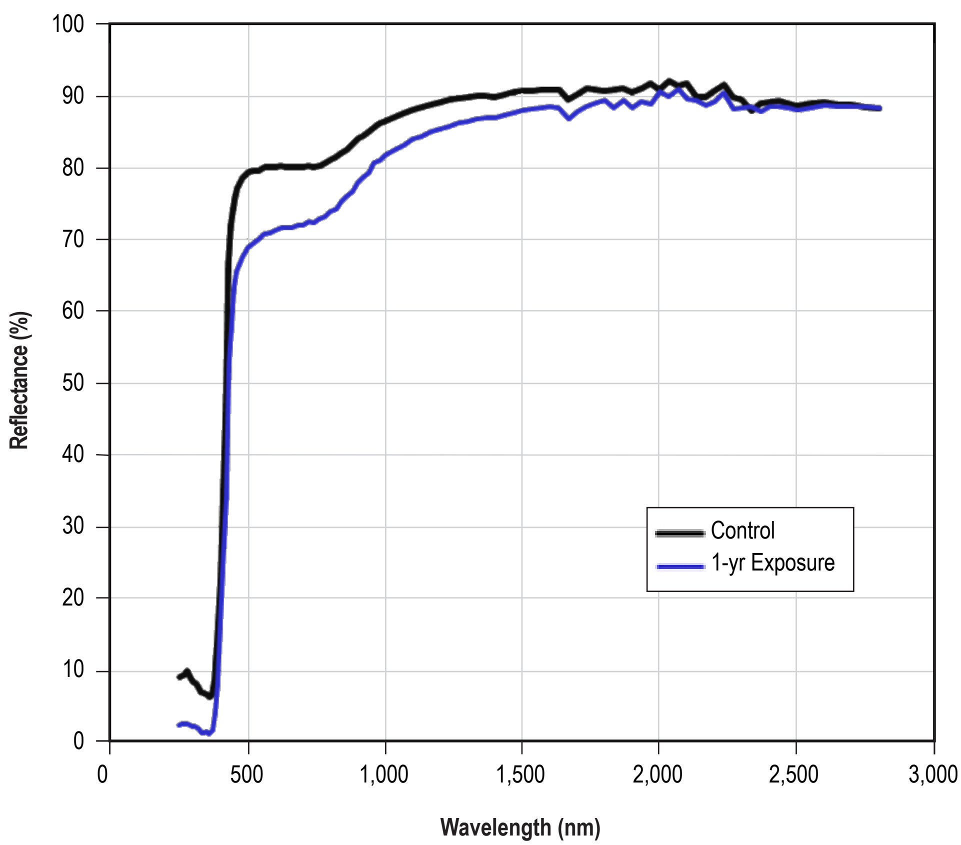

Figure 9. Reflectance of aluminized CP1 with polymer side exposed.
Reflectance is of VDA as second surface, through CP1.

In 2005, a solar sail of 2-µm CP1 with VDA was successfully tested at GRC.[6] In addition to launch vibration and ascent vent tests, the 20-m^2 sail was deployed under high vacuum and 16 ºC temperature in the Plum Brook Station test chamber. These tests led to the successful Nanosail-D2 mission[7] 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.

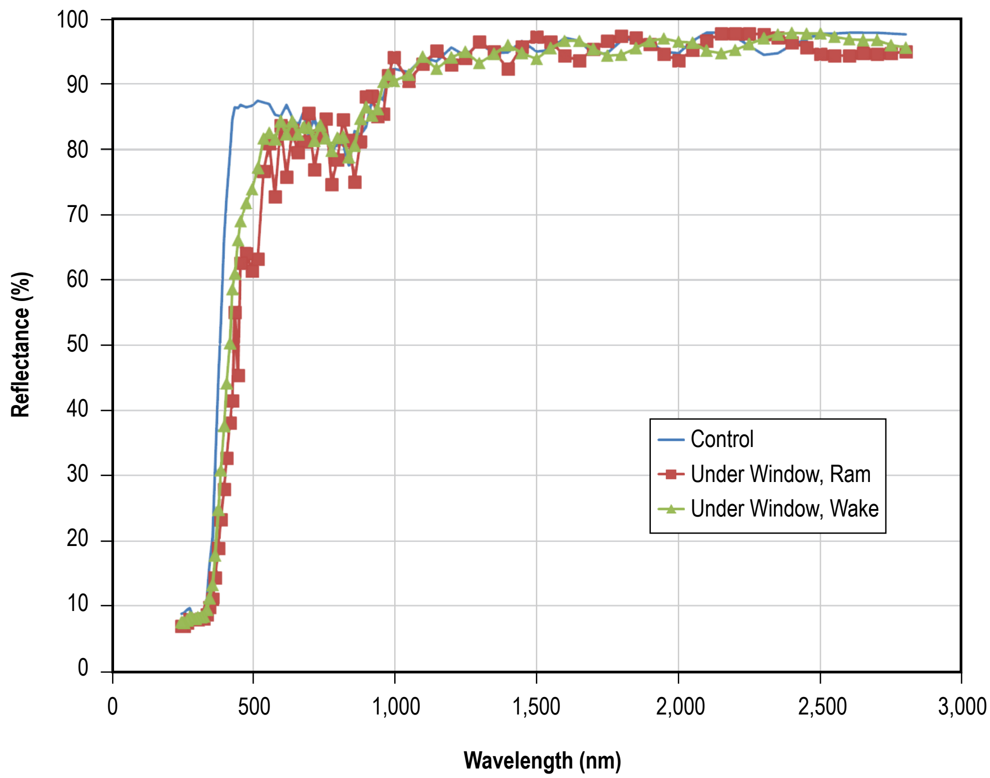

Figure 10. Reflectance of 2-µm CP1 with VDA.

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 1.3×10^{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.

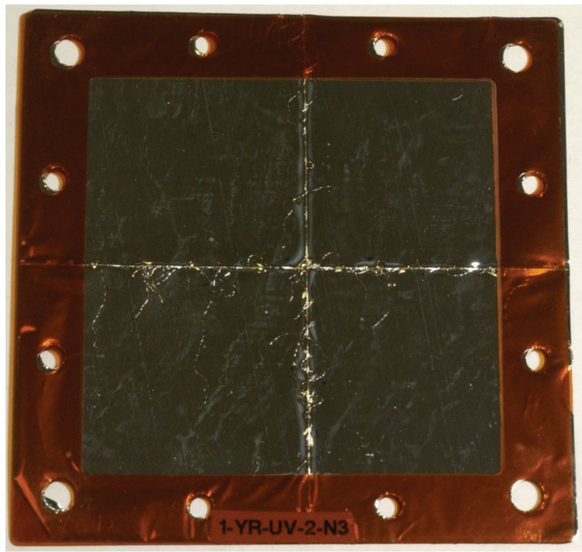

Figure 11. MISSE-1 CP1 with VDA and SiO_x coatings and Kevlar ripstop.

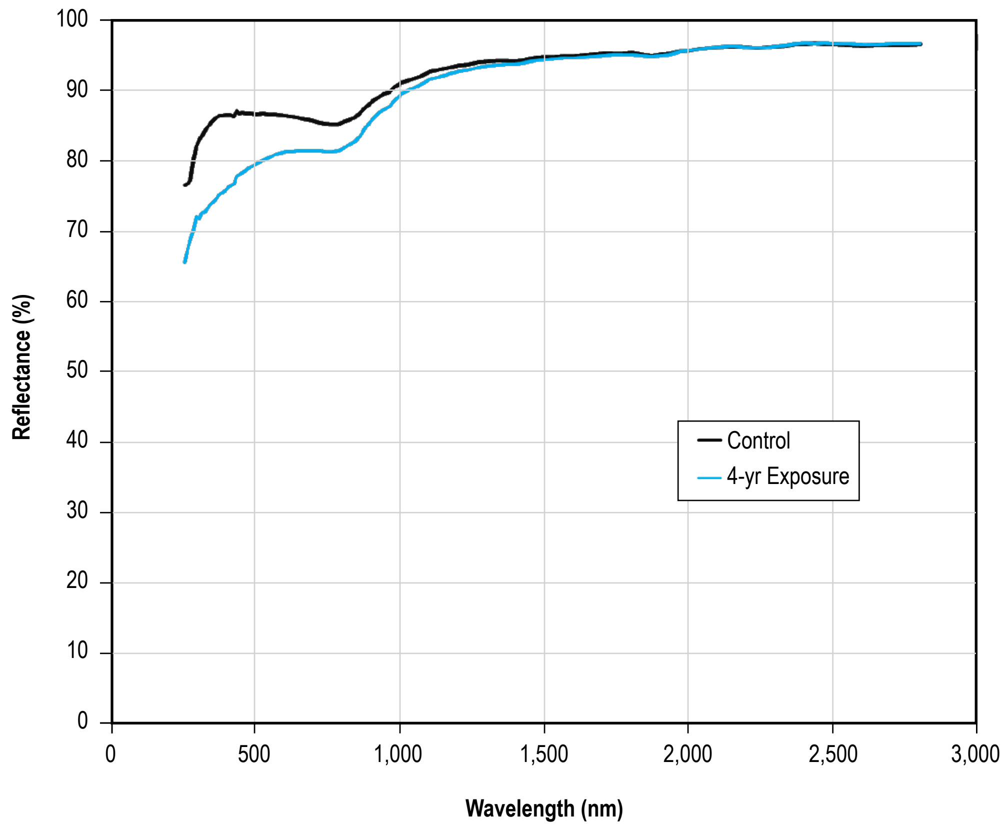

Figure 12. Reflectance of MISSE-1 CP1 with VDA, SiO_x, and ripstop.

A thicker sample without the ripstop was flown on the ram-facing side of MISSE-4 and exposed to 2.1×10^{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.

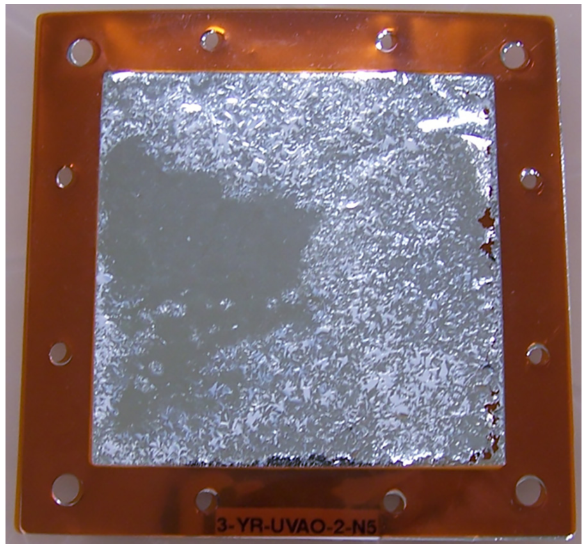

Figure 13. MISSE-4 CP1 with VDA and SiO_x coatings.

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 2×10^{21} oxygen atoms/cm^2. Mass loss for this sample indicates an AO erosion yield of 9×10^{-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).

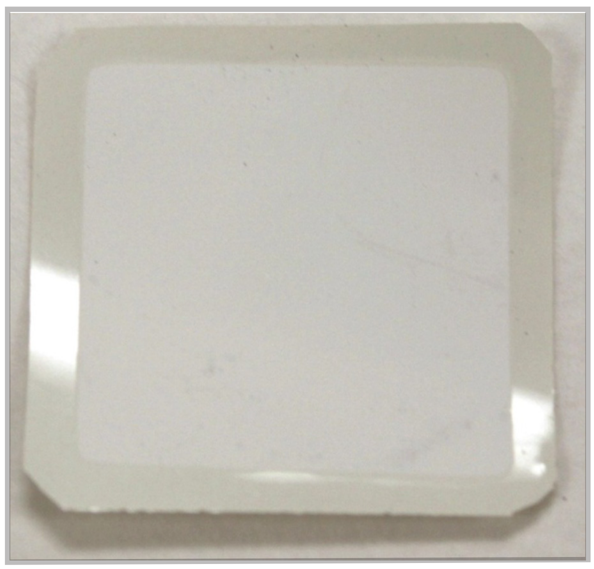

Figure 14. MISSE-6A conductive white CP1.

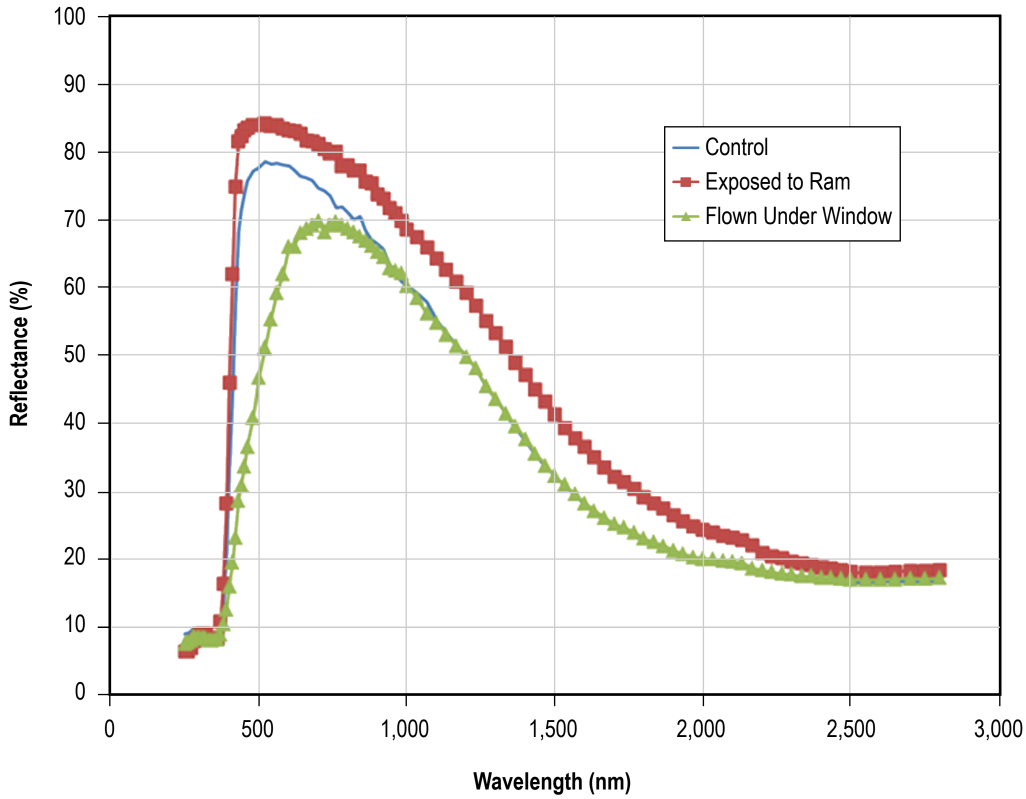

Figure 15. Reflectance of MISSE-6 conductive white CP1.

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.

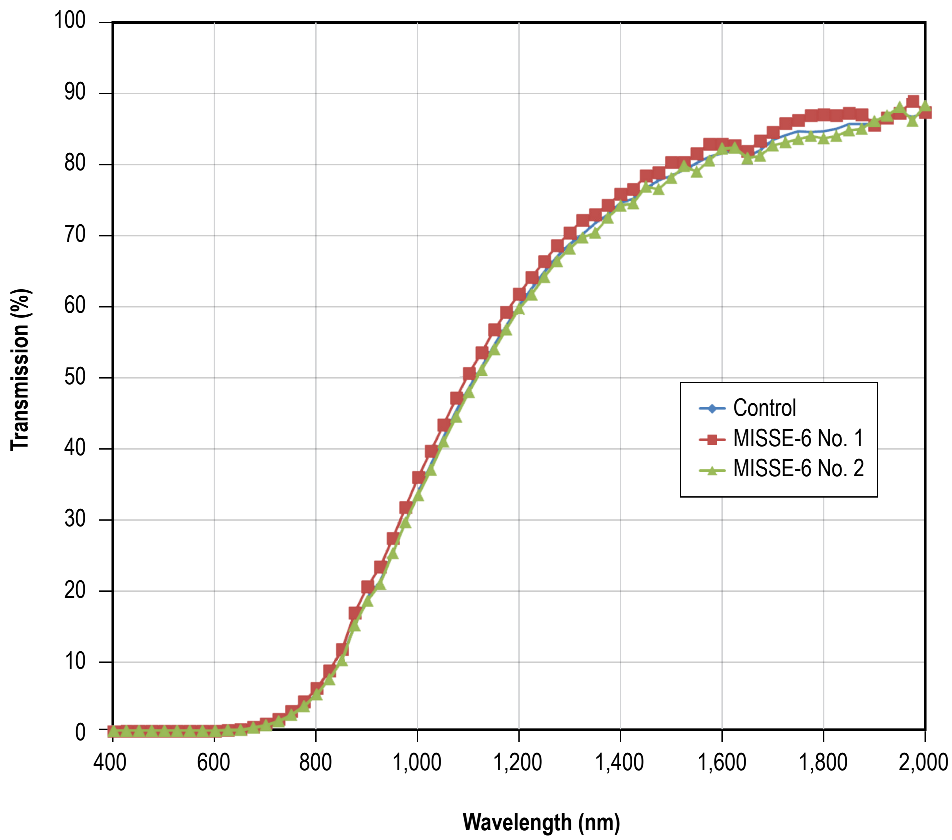

Figure 16. Visible to near-infrared transmission of infrared transparent CP1.

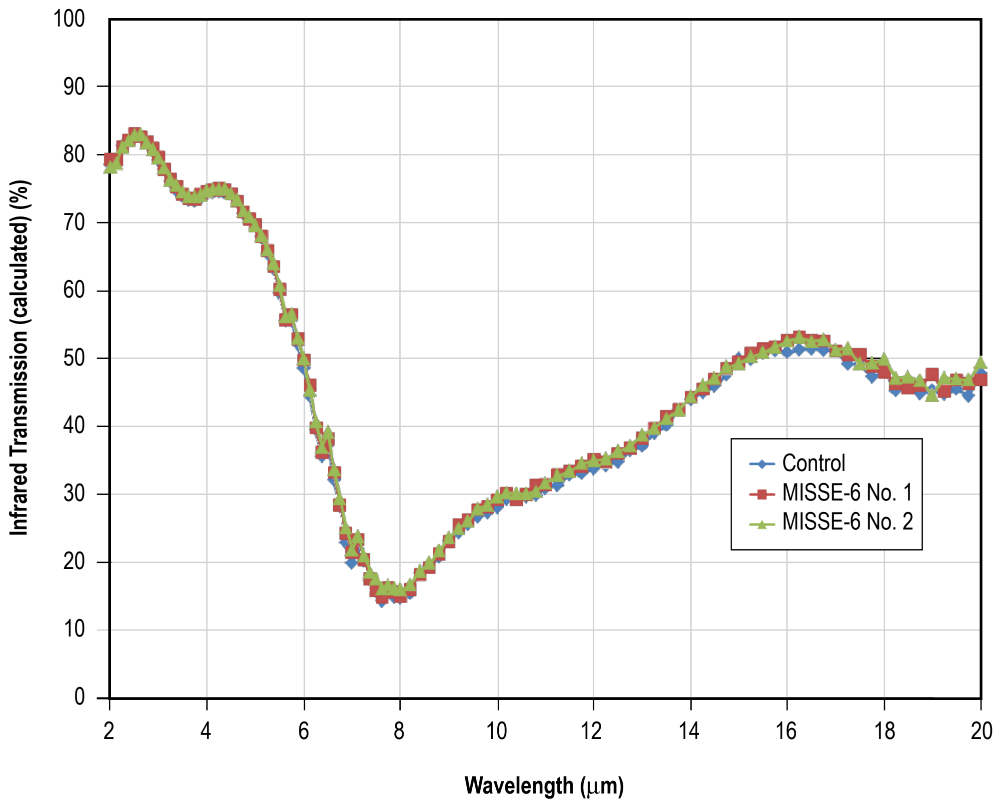

Figure 17. Calculated infrared transmission for infrared transparent CP1.

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 6.5×10^{-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 2.6×10^{-26} cm^3/atom. Figure 19 shows the reflectance curve for the conductive EP2550.

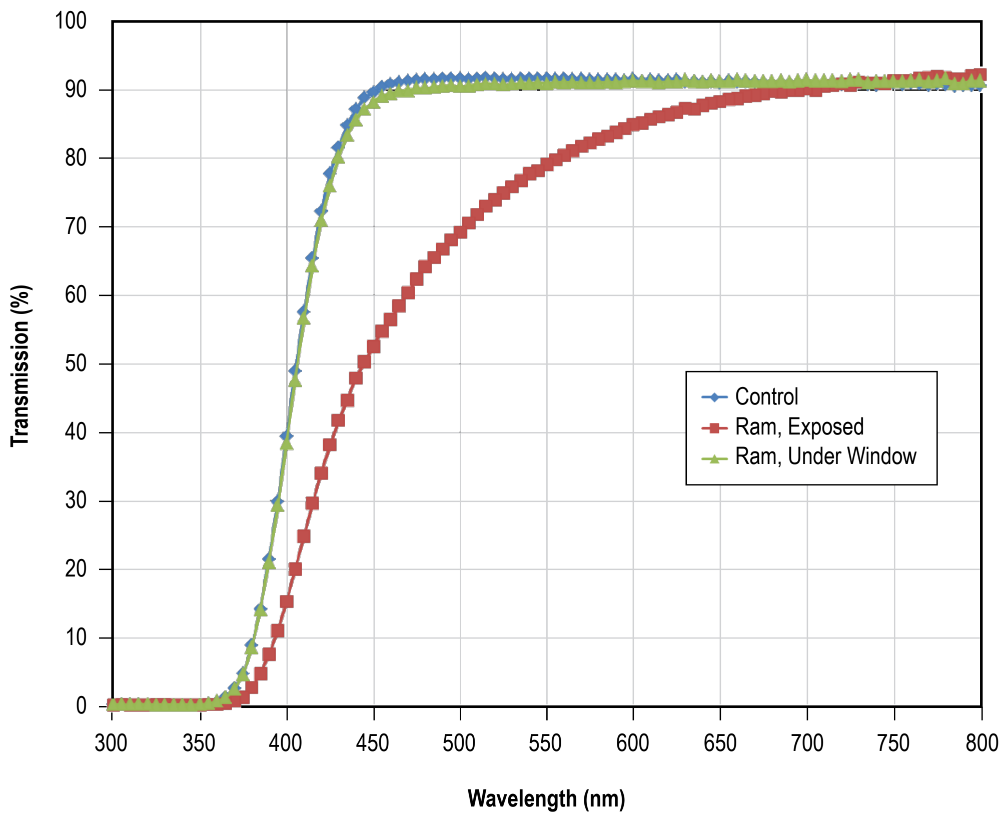

Figure 18. Transmission of MISSE-6 EP2550 samples.

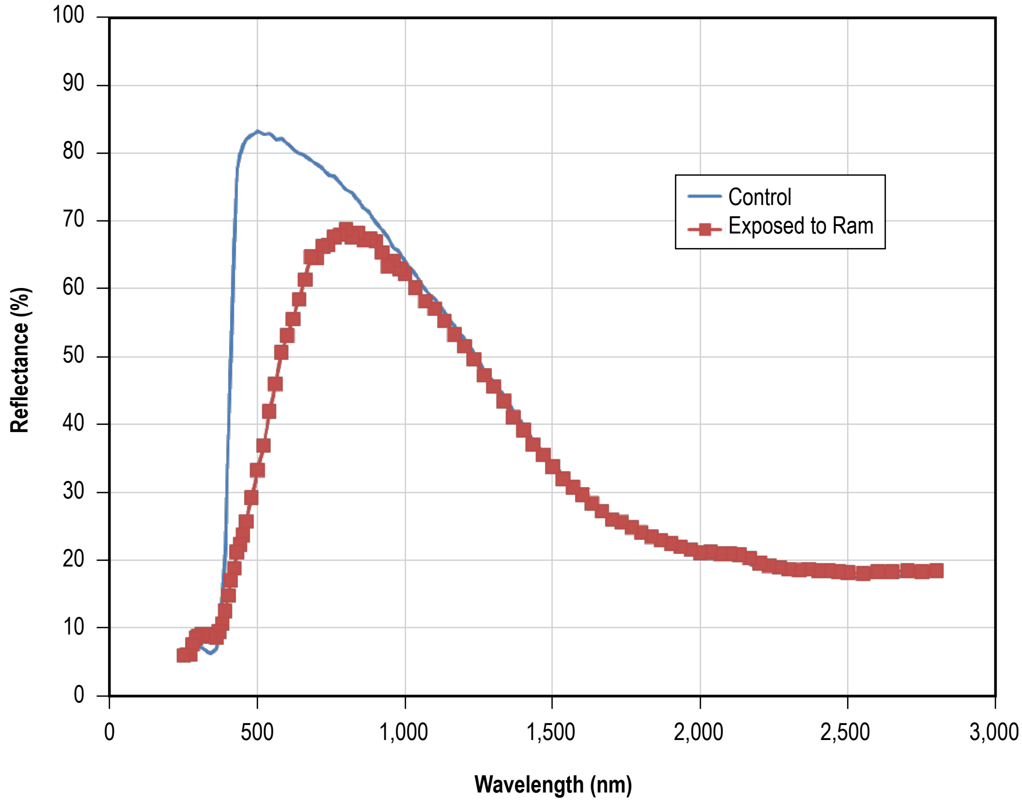

Figure 19. Reflectance of MISSE-6A conductive EP2550.

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.

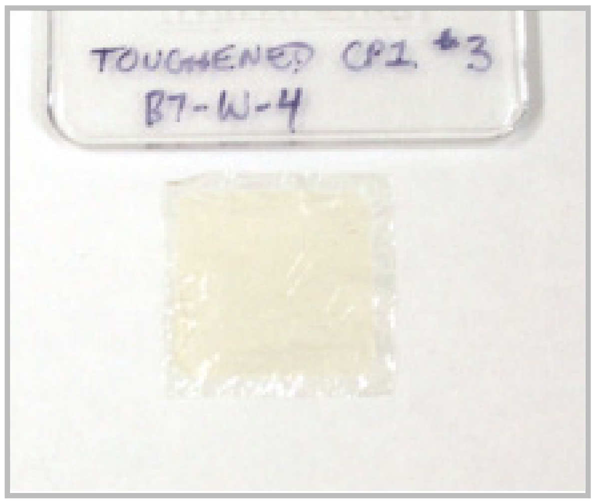

Figure 20. Toughened CP1 flown on MISSE-7B wake side.

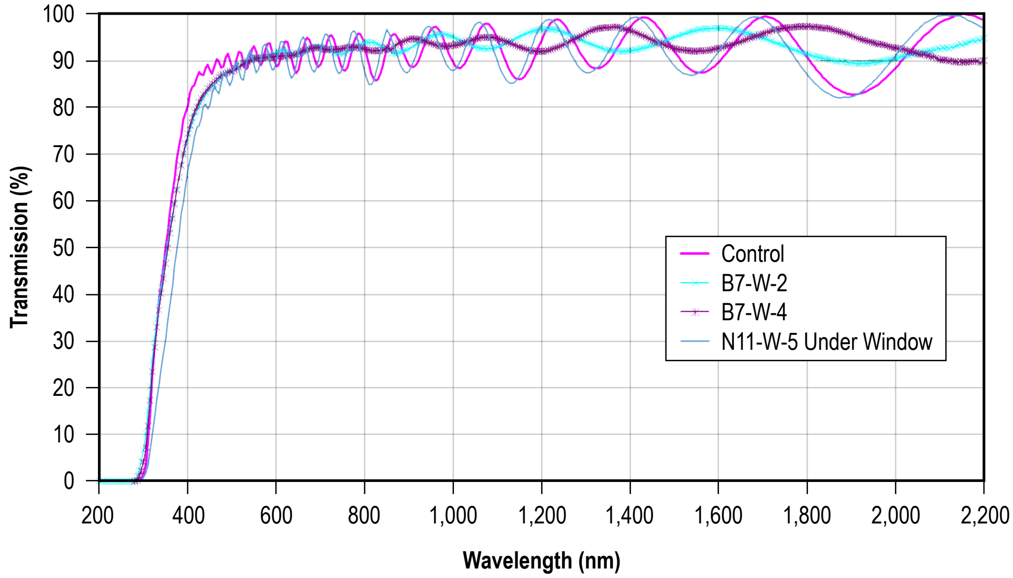

Figure 21. Transmission of toughened CP1.

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 8.4×10^{-26} cm^3/atom; the transmission is shown in figure 22.

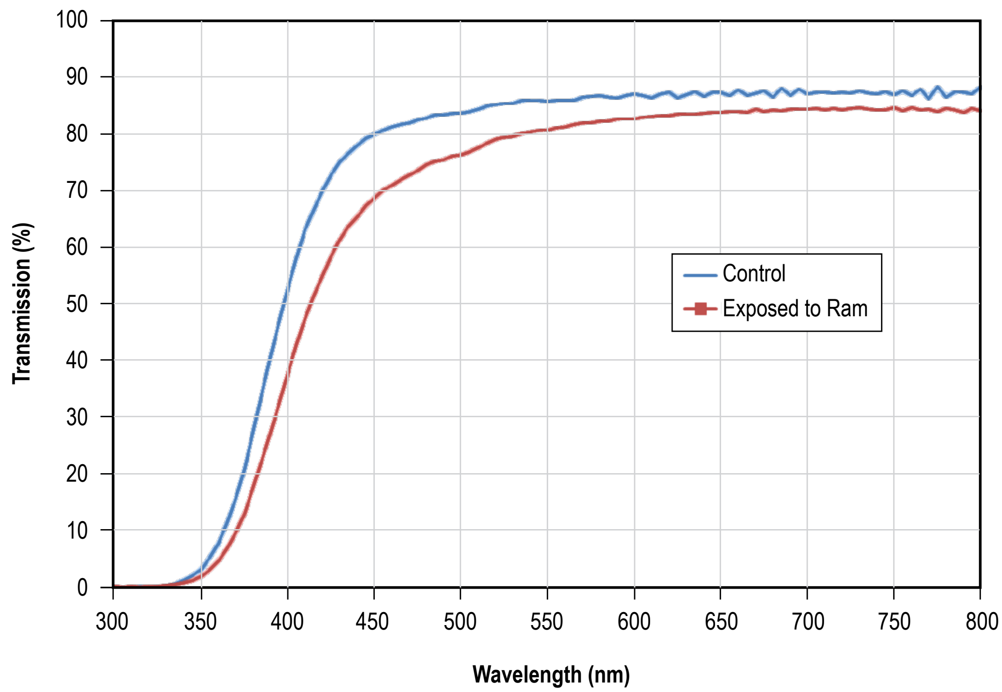

Figure 22. Transmission of AO-resistant CP1.

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.

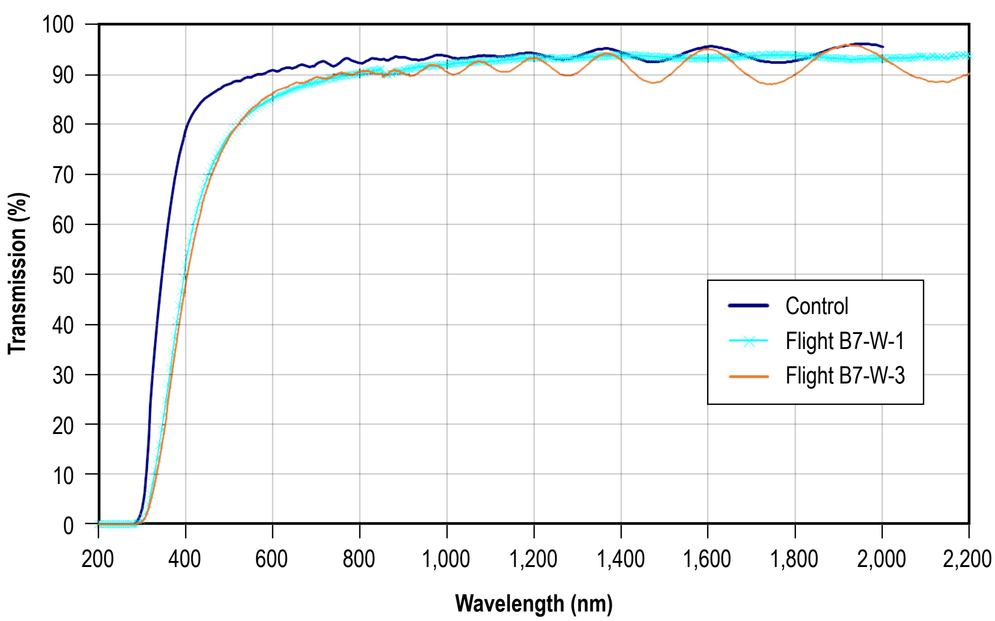

Figure 24. Transmission of toughened CORIN® XLS.

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

1. de Groh, K.K.; Banks, B.A.; McCarthy, C.E.; et al.: “MISSE PEACE Polymers Atomic Oxygen Erosion Results,” NASA/TM—2006–214482, NASA Glenn Research Center, Cleveland, OH, 25 pp., November 2006.

2. Materials and Processes Technical Information System, <http://maptis.nasa.gov/>, July 2014.

3. Pippin, G.: “Summary Status of MISSE-1 and MISSE-2 Experiments and Details of Estimated Environmental Exposures for MISSE-1 and MISSE-2,” Paper AFRL-ML-WP-TR-2006-4237, United Technology Corporation, Dayton, OH, Final Report for the Air Force Research Laboratory, June 24, 2002–July 31, 2006, July 2006.

4. Finckenor, M.M.; Pippin, G.; and Kinard, W.H.: “Estimated Environmental Exposures for MISSE-3 and MISSE-4,” Paper presented at National Space & Missile Material Symposium, Henderson, NV, June 23–27, 2008.

5. de Groh, K.K.; Finckenor, M.M.; Minton, T.; et al.: “Post-Flight Analysis of Selected Fluorocarbon and Other Thin Film Polymer Specimens Flown on MISSE-5,” Paper presented at National Space & Missile Materials Symposium, Keystone, CO, June 25–29, 2007.

6. Johnson, L.; Young, R.; Montgomery, E.; and Alhorn, D.: “Status of solar sail technology within NASA,” *Adv. Space Res.*, Vol. 48, No. 11, pp. 1687–1694, doi: 10.1016/j.asr.2010.12.011, December 2011.

7. Alhorn, D.C.; Casas, J.P.; Agasid, E.F.; et al.: “NanoSail-D: The Small Satellite That Could!,” Paper SSC11-VI-1, *Proc. AIAA/USU Conf. Small Satellites*, Logan, UT, August 8–11, 2011.

8. de Groh, K.K.; Banks, B.A.; Guo, A.; et al.: “MISSE 6 Polymers Atomic Oxygen Erosion Data,” Paper presented at National Space & Missile Materials Symposium, Scottsdale, AZ, June 28–July 1, 2010.

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	01–03–2015
2. REPORT TYPE	Technical Memorandum
3. DATES COVERED *(From - To)*	
4. TITLE AND SUBTITLE	Analysis of Fluorinated Polyimides Flown on the Materials International Space Station Experiment
5a. CONTRACT NUMBER	
5b. GRANT NUMBER	
5c. PROGRAM ELEMENT NUMBER	
5d. PROJECT NUMBER	
5e. TASK NUMBER	
5f. WORK UNIT NUMBER	
6. AUTHOR(S)	M.M. Finckenor, L. Rodman,* and B. Farmer*
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)	George C. Marshall Space Flight Center Huntsville, AL 35812
8. PERFORMING ORGANIZATION REPORT NUMBER	M–1394
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	National Aeronautics and Space Administration Washington, DC 20546–0001
10. SPONSORING/MONITOR'S ACRONYM(S)	NASA
11. SPONSORING/MONITORING REPORT NUMBER	NASA/TM—2015–218205
12. DISTRIBUTION/AVAILABILITY STATEMENT	Unclassified-Unlimited Subject Category 27 Availability: NASA STI Information Desk (757–864–9658)
13. SUPPLEMENTARY NOTES	Prepared by the Materials & Processes Laboratory, Engineering Directorate *NeXolve Corporation, Huntsville, AL

14. 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.

15. SUBJECT TERMS

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

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON / 19b. TELEPHONE NUMBER *(Include area code)*
U	U	U	UU	36	STI Help Desk at email: help@sti.nasa.gov STI Help Desk at: 757–864–9658

National Aeronautics and
Space Administration
IS20
George C. Marshall Space Flight Center
Huntsville, Alabama 35812