

# The Materials On International Space Station Experiment (MISSE): First Results From MSFC Investigations

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Hundreds of material samples were passively exposed to the space environment for nearly four years as part of the Materials on *International Space Station* Experiment (MISSE). The experiment was planned for one year of exposure, but its return was delayed by the *Columbia* accident and subsequent grounding of the Space Shuttle fleet. The experiment was attached externally to the Quest Airlock. Atomic oxygen fluence and ultraviolet radiation dose varied across the experiment because of shadowing and space station orientation. Over a hundred meteoroid/space debris impacts were found. Many polymer film samples were completely eroded by atomic oxygen. Some particulate contamination was noted, but black light inspection and the transmission measurements of magnesium fluoride windows indicated that molecular contaminant deposition was limited. Optical property changes in thermal control materials are discussed.

## I. Introduction

In August 2001 during the STS-105 mission to the *International Space Station* (ISS), astronauts Daniel Barry and Patrick Forrester deployed two suitcases of materials samples to be passively exposed to the space environment. These were the Materials on *International Space Station* Experiment, or MISSE, -1 and -2 payloads. These suitcases were attached to the ISS on the Quest airlock (fig. 1), where they were exposed to atomic oxygen (AO), ultraviolet (UV) radiation, particulate radiation, thermal cycling, and the induced environment of an active space station.

The MISSE program is a combined effort between the Air Force Research Laboratory, NASA, Boeing Phantom Works, and other aerospace companies, to study material behavior in low Earth orbit. Langley Research Center was responsible for the suitcase, or Passive Experiment Carrier (PEC), design, manufacture, sample integration, and qualification for spaceflight. The PEC design was proven earlier with the four Mir Environmental Effects Payload experiments<sup>1</sup>. MISSE-1 and -2 have completed their mission, MISSE-5 is an active experiment currently deployed on ISS, and MISSE-3 and -4 are scheduled for deployment in 2006.

Marshall Space Flight Center (MSFC) provided not only material samples, but also supported thermal vacuum bakeouts and determination of outgassing properties of materials through either MSFC-SPEC-1443 or ASTM-E-595. MSFC also performed pre-flight characterizations of mass, solar absorptance, infrared emittance, and optical transmission for a number of samples.

The planned exposure for MISSE-1 and -2 was nominally one year. After the *Columbia* accident, the shuttle fleet was grounded for two and a half years. MISSE-1 and -2 were not retrieved until STS-114, after nearly four years of exposure. Astronauts Steve Robinson and Soichi Noguchi retrieved MISSE-1 and -2, and later deployed MISSE-5.

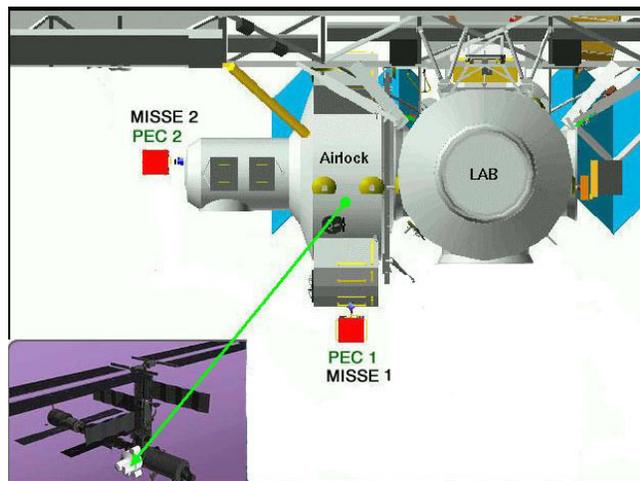


Figure 1. Locations of MISSE -1 and -2 on ISS

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At the time of publication, full evaluation of all the MISSE samples was not complete. However, some early MISSE results may be useful for current spacecraft designs, such as the Crew Launch Vehicle, the Crew Exploration Vehicle, and the James Webb Space Telescope.

## II. Environment

The MISSE suitcases were intended to fly with one face in the ram direction and one face in the wake direction. This would expose one set of samples to AO attack, while the samples in the wake would be protected and see degradation due only to UV radiation. However, *ISS* flew in local vertical, local horizontal (LVLH) attitudes, both X-axis in Velocity Vector and Y-axis in Velocity Vector, and X-axis perpendicular to orbital plane (XPOP) attitude during the MISSE exposure. This means that the “wake” face of MISSE-1 was exposed to AO, which Kapton HN erosion indicated was approximately  $1.1 \times 10^{20}$  atoms/cm<sup>2</sup> fluence. A Russian solar array blocked the “wake” face of MISSE-2 from AO erosion, but this has not yet been confirmed by polymer sample mass measurements. Here after, the “ram” faces of MISSE-1 and -2 refer to the sides that received the greater AO fluence, and the “wake” faces refer to the sides that received the lesser amount of AO.

Some uncertainty in the MISSE environment is also due to shielding by the Quest airlock. Mass loss of polymers across the suitcase indicates 20-25% variation in AO erosion, with those closest to the airlock showing the least erosion. This variation also holds true for UV exposure, as can be seen in fig. 2.

MSFC flew UV diodes for measuring the solar exposure on both the wake face of MISSE-1 and the ram face of MISSE-2. Photoelectron production in the diode created a current which was then collected by a coulometer, measuring the integrated current over the entire MISSE exposure. These UV sensors were optimized for one to 1.5 year exposure and were completely saturated after nearly four years in space. The measured UV dose was 1400 equivalent sun-hours (ESH), but the actual dose is at least 5000 ESH, as determined from modeling.



**Figure 2. Shadowing of MISSE-1 by the airlock**



**Figure 3. MISSE-2, Ram face**

Both ram faces of MISSE-1 and -2 received a minimum of  $4.2 \times 10^{21}$  atoms/cm<sup>2</sup> of AO, as determined by the complete erosion of 5 mils of Kapton HN and other thin polymer films. The maximum possible AO fluence for MISSE at the *ISS* altitude for 47.5 months, assuming full ram exposure with no shadowing, is  $1.17 \times 10^{22}$  atoms/cm<sup>2</sup>, based on MSIS-86 model predictions. The true exposure must be somewhere between these values. Erosion of 2-mil and 5-mil thick FEP Teflon with silver/Inconel metallization from tray E7 (fig. 3) indicated a fluence of  $4.5 \times 10^{21}$  atoms/cm<sup>2</sup>, assuming an AO reactivity of  $0.35 \times 10^{-24}$  cm<sup>3</sup>/atom. However, this FEP Teflon reactivity was determined on samples from the Long Duration Exposure Facility (LDEF). There is some discussion as to whether that same AO reactivity is a good assumption, given the synergistic role of UV in fluoropolymer degradation. AO reactivity for FEP Teflon determined from short exposures on Shuttle flights<sup>2</sup> was less than  $0.05 \times 10^{-24}$  cm<sup>3</sup>/atom. This is much lower than that for LDEF because of the minute

amount of synergistic UV to aid in breaking molecular bonds. Pippin et al<sup>3</sup> state that the AO reactivity of metallized FEP Teflon can be approximated as a semilog function of solar exposure, as follows:

$$Re = 0.0555 \ln(\text{ESH}) - 0.184 \quad (1)$$

where ESH is the solar exposure in equivalent sun hours. Assuming the MISSE samples received on the order of 5000 ESH, the AO reactivity may be on the order of  $0.29 \times 10^{-24} \text{ cm}^3/\text{atom}$ . This would indicate an AO fluence of  $5.4 \times 10^{21} \text{ atoms/cm}^2$ .

Mass loss of the Triton Oxygen Resistant (TOR) polymer from tray E8 indicated a fluence of  $6.5$  to  $6.8 \times 10^{21} \text{ atoms/cm}^2$ , using AO reactivity determined from the Passive Optical Sample Assembly (POSA)-1 flight experiment and ground exposures in the 5 eV MSFC Atomic Oxygen Beam Facility.

Radiation dosimeters were flown in a number of locations on MISSE, but the results were not available at time of publication.

The samples were exposed to a thermal environment of mostly  $-40^\circ\text{C}$  to  $+40^\circ\text{C}$ . MISSE-1 and -2 went through approximately 22,800 thermal cycles.

The experiment suitcases were surveyed for meteoroid and orbital debris impacts. 119 impacts were found on the sample trays and baseplates, and 16 impacts were found on the sides of the PECS during de-integration at Langley. Another seven impacts were found later. The largest impact found was approximately 3 mm in diameter. Impacts on the samples and baseplates were almost uniformly distributed between the ram faces and wake faces (table 1). This is very different from the gravity-gradient stabilized LDEF, where the ram face had an order of magnitude more impacts than the wake face.

Table 1. Distribution of Micrometeoroid/Space Debris Impacts

Location	# Impacts
MISSE-1 Ram face	34
MISSE-1 Wake face	33
MISSE-2 Ram face	29
MISSE-2 Wake face	30

Particulate contamination was more of a concern than molecular contamination. Particulates were mainly produced by mechanical failure of some of the eroded polymers, though silver oxide particles were observed. Visual and black light inspection indicated very little molecular contamination, with only some deposition in the vicinity of a composite facesheet-aluminum honeycomb core sandwich (fig. 4). Magnesium fluoride windows indicated little change in transmission.



Figure 4. Deposition on MISSE baseplate from composite sub-experiment.

### III. Results

Magnesium fluoride windows were used to expose candidate solar sail materials to UV radiation while blocking all AO. Transmission was measured before and after flight to determine the level of molecular contamination. The windows from tray E7 had very little change in transmission (fig. 5), and some of the windows from tray E8 showed a decrease in transmission, particularly in the shorter wavelengths (fig. 6). This change in transmission is comparable to some windows from the POSA-1 flight experiment that had 250 – 300 Å of SiOx contamination<sup>4</sup>. However, as of publication, the contaminant film has not been positively identified as SiOx or some other chemical composition.

Solar absorptance measurements of AZ93 thermal control coating samples support the observation of little molecular contamination on MISSE. This coating, composed of zinc oxide in a potassium silicate binder, can absorb contamination and subsequently darken<sup>5</sup>. While AO can remove hydrocarbon contamination, AO will convert silicone contamination into a permanent silicate layer on the material surface. The POSA-1 flight experiment had zinc oxide/potassium silicate thermal control coating (IITRI Z93P) that darkened from a solar absorptance of 0.160 to 0.228 during 18 months exposure in the presence of silicone contamination<sup>4</sup>. Similar coatings on MISSE, however, maintained a pristine white color (fig. 7) and had solar absorptance measurements of 0.15, within instrument error of pre-flight measurements (fig. 8).

Triton Systems, Inc. of Chelmsford, MA has developed a number of AO-resistant polymers under a small business innovative research (SBIR) contract to MSFC. TOR polymer film of 2 mil thickness performed well, as did the low modulus version TOR-LM. Both TOR and TOR-LM were eroded, but the AO reactivity was  $1.6 \times 10^{-25}$  cm<sup>3</sup>/atom or less, assuming the minimum AO fluence of  $4.2 \times 10^{21}$  atoms/cm<sup>2</sup> previously mentioned. COR film, a clear version of TOR, yellowed but was intact. C-COR and C-TOR-LM films were developed by Triton as conductive versions of COR and TOR-LM, mainly for electrostatic dissipative purposes. 1.5-mil thick C-COR had a cracked appearance, while the C-TOR-LM film of the same thickness was mostly eroded through. AO reactivities for the conductive films were on the order of  $4$  to  $5 \times 10^{-25}$  cm<sup>3</sup>/atom.

Of interest to the *ISS* program was the performance of beta cloth, a woven fiberglass mat impregnated with Teflon, on MISSE. Aluminized Chemfab 500F beta cloth is used as an outer cover on multi-layer insulation (MLI) blankets on *ISS*. Chemfab 500F is similar to the Chemfab 250F used in the Space Shuttle and Spacelab MLI blankets, except that the silicone usually added for flexibility is omitted. Samples of aluminized beta cloth from the same batch as *ISS* MLI blankets were flown on both the ram and wake faces of MISSE. Samples of beta cloth with slightly larger glass fibers, referred to as “beta cloth plus”, and Chemfab 500F beta cloth without the aluminization were also flown. All of the beta cloth samples were slightly darkened by the exposure (fig. 9). Solar absorptances for control and flight samples are given in Table 2. All measurements were made with an AZ Technology Laboratory Portable Spectroreflectometer (LPSR) with a black background.

Table 2. Solar Absorptance of MISSE Beta Cloth

Material ID	Material Type	Environmental Exposure	Solar Absorptance
Control	ISS Alum. Beta	None	0.343
2-E7-6	ISS Alum. Beta	High AO, UV	0.381
2-E7-14	ISS Alum. Beta	High AO, UV	0.368
2-E11-10	ISS Alum. Beta	Low AO, High UV	0.385
2-E11-16	ISS Alum. Beta	Low AO, High UV	0.384
Control	Beta Plus	None	0.358
2-E7-19	Beta Plus	High AO, UV	0.390
2-E7-29	Beta Plus	High AO, UV	0.383
2-E11-18	Beta Plus	Low AO, High UV	0.403
2-E11-29	Beta Plus	Low AO, High UV	0.391
Control	Chemfab 500F	None	0.355
2-E11-17	Chemfab 500F	Low AO, High UV	0.378
2-E11-20	Chemfab 500F	Low AO, High UV	0.382

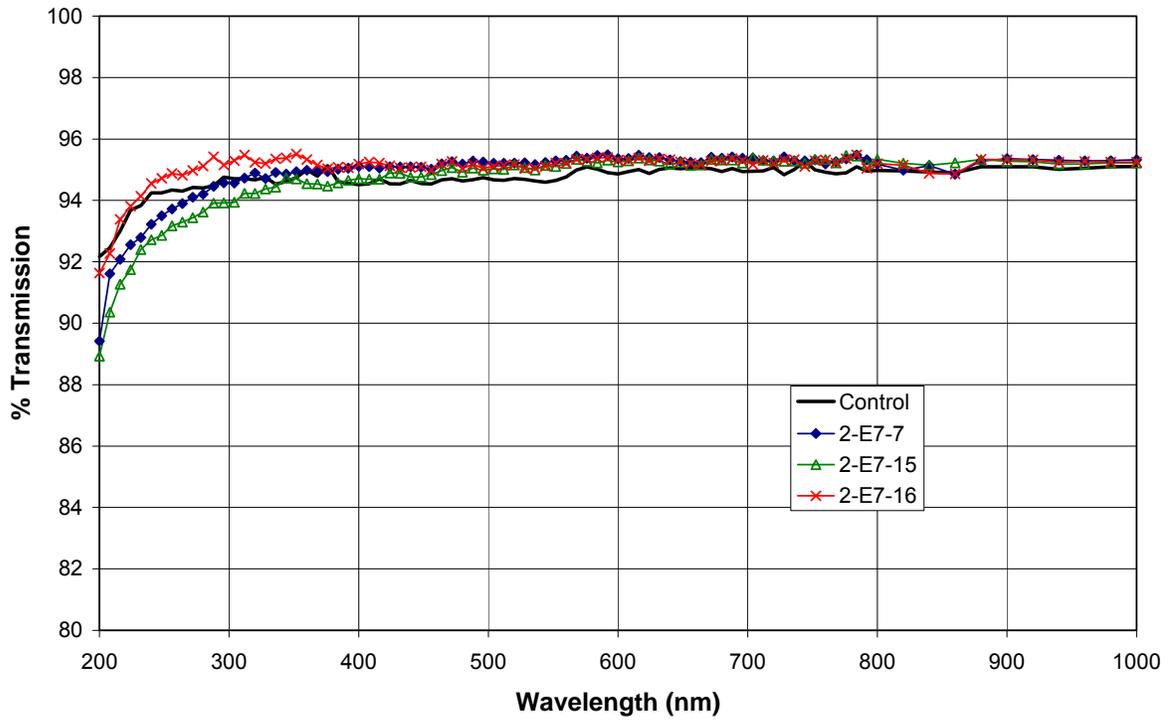


Figure 5. Transmission of magnesium fluoride windows on tray E7, MISSE-2 ram face

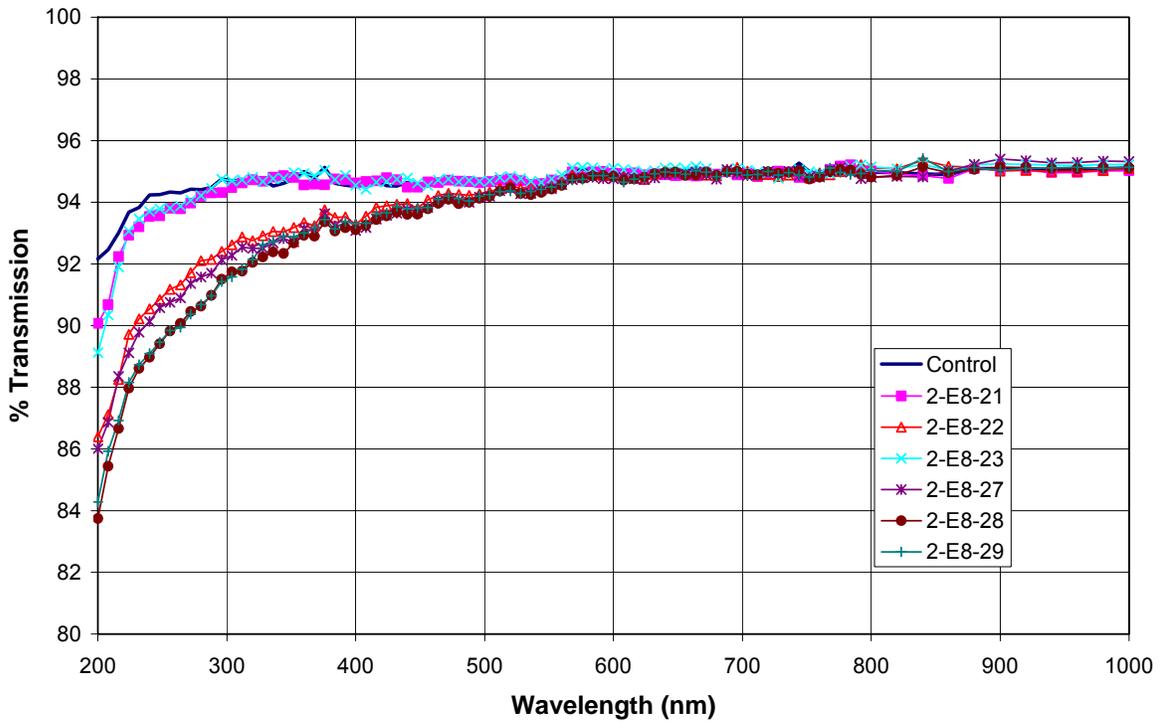


Figure 6. Transmission of magnesium fluoride windows on tray E8, MISSE-2 ram face

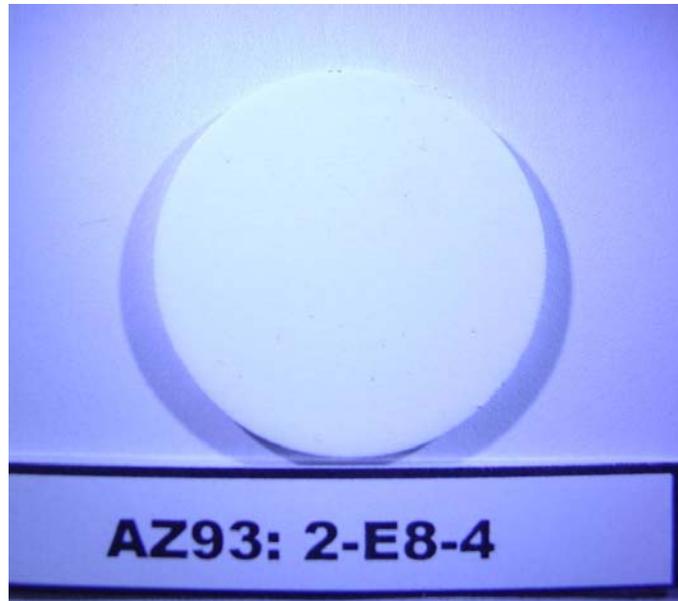


Figure 7. AZ93 thermal control coating from MISSE-2, ram face

### MISSE AZ93

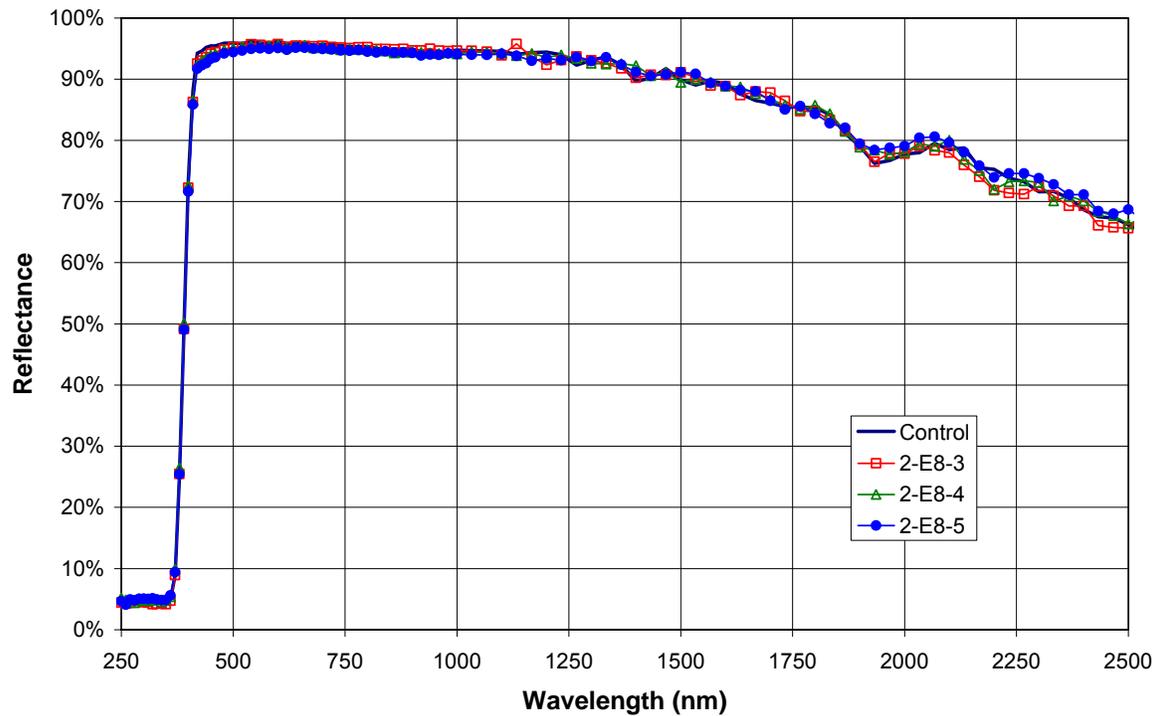


Figure 8. Reflectance measurements of AZ93 thermal control coating exposed on MISSE-2, ram face

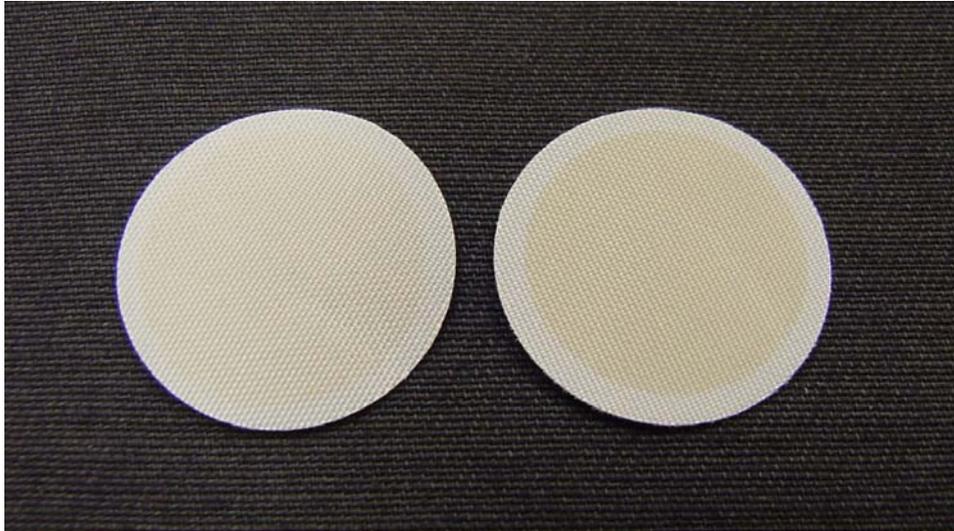


Figure 9. Aluminized Chemfab 500F from same batch as ISS MLI blankets (a) ram face of MISSE-2 (b) wake face of MISSE-1

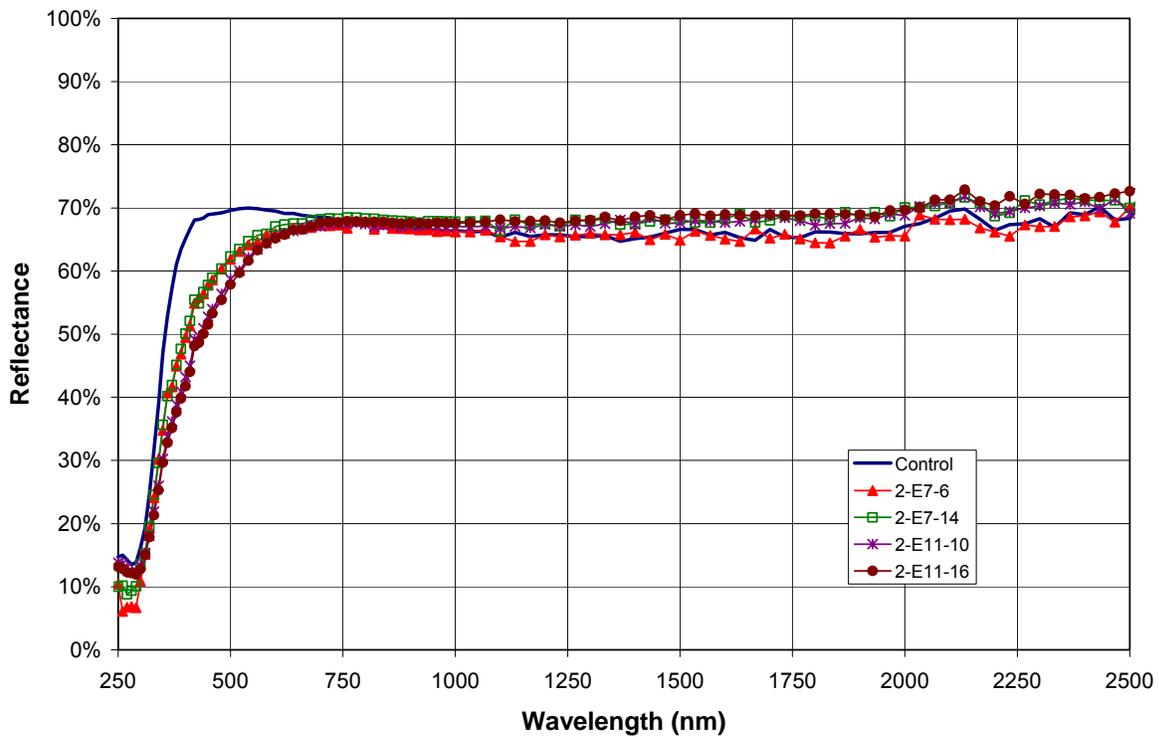


Figure 10. Reflectance measurements of aluminized Chemfab 500F (ISS batch), exposed on MISSE-2 ram face (E7) and MISSE-1 wake face (E11).

Kynar shrink tubing is used on *ISS*. This was a classic example of UV darkening and AO counter-effects, depending on the location on MISSE. Kynar on the wake face with little AO darkened considerably (fig. 11), while Kynar on the ram face darkened only slightly. AO erosion on the ram-facing Kynar was evident. Reflectance curves for these samples are shown in figure 12.

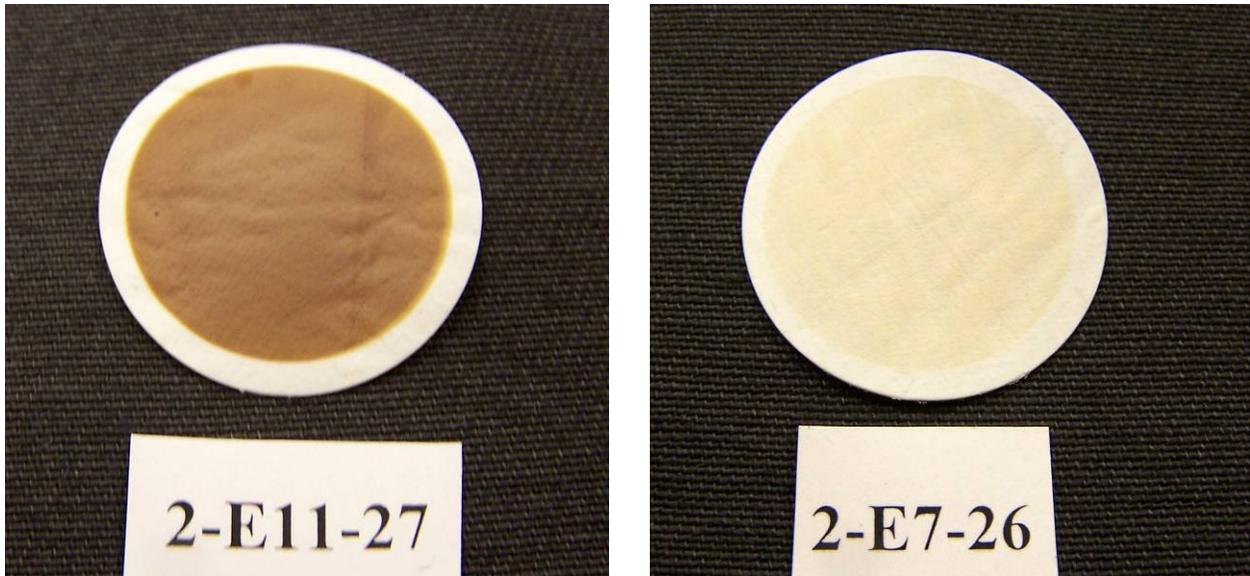


Figure 11. (a) Kynar with little AO exposure and UV darkening (b) AO-exposed Kynar with slight darkening

### MISSE Kynar

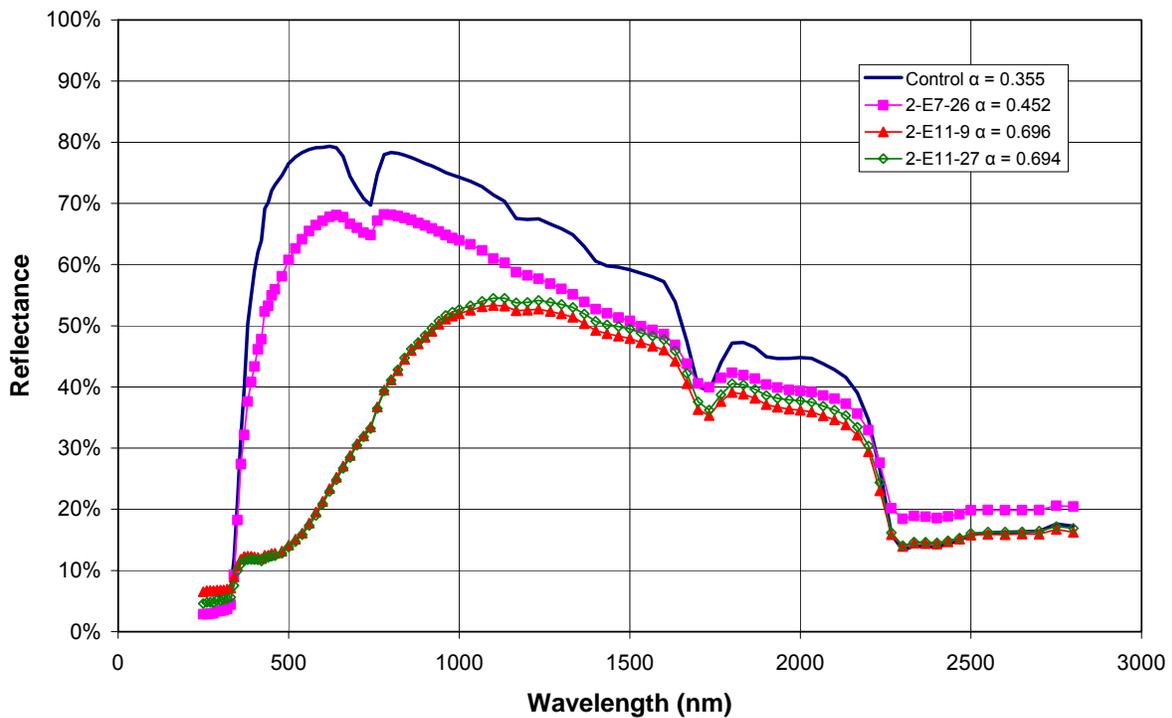


Figure 12. LPSR measurements of Kynar from MISSE-1, wake face (E11) and MISSE-2, ram face (E7)

#### IV. Discussion and Conclusions

The space environment exposure did not appear to have a significant effect on the optical properties of AZ93 zinc oxide/potassium silicate thermal control coating on the ram face of MISSE-2. Magnesium fluoride windows, particularly on the E7 tray, maintained good transmission down to 200 nm wavelength. Some darkening was noted on various types of beta cloth, both on the ram and wake faces. Many of the thin polymer films exposed on the ram faces were completely eroded away, with the exception of some fluoropolymers and the TOR and COR polymeric materials developed by Triton Systems.

Some science on MISSE was lost due to the extended mission, but, particularly for the thermal control materials, the additional AO and UV was helpful in evaluating long term durability. Lack of widespread molecular contamination on MISSE gives confidence in using *ISS* as a scientific platform for materials study, though the locations of future flight experiments should be analyzed for line-of-sight to possible contamination sources, such as engine plumes, vents, etc. MISSE's cleanliness gives us confidence in the contamination control methods used, especially materials selection and thermal vacuum bakeout criteria. While some may point to the lack of Space Shuttle flights for over two years as the reason for the low amount of contamination, there were a total of 14 Progress flights, 9 Soyuz flights, and 6 Space Shuttle flights to *ISS* during the time MISSE-1 and -2 were deployed. The MISSE experiments were attached to the Quest Airlock, which had been in space barely a month as of August 2001. New hardware added to *ISS* between August 2001 and July 2005 included the S0, S1, and P1 Trusses, the Canadarm2 and Mobile Transporter, the Strela boom crane, and the Pirs docking module. This hardly constitutes a quiescent exposure to space.

Investigations are continuing for a number of samples not mentioned in this paper. Fourier transform infrared (FTIR) spectroscopy, ellipsometry, and other techniques will be used to study contaminant depositions. Vacuum UV transmission in the 120 to 200 nm wavelength band will be measured for the magnesium fluoride windows.

#### Acknowledgments

The success of the MISSE experiment would not have been possible without the significant contributions of Dr. Michael Stropki of the Air Force Research Laboratory, Dr. William Kinard of Langley Research Center, and Dr. Gary Pippin and Dr. Suzanne Woll of Boeing Phantom Works. The author gratefully acknowledges Rachel Kamenetzky for her pre-flight work with MISSE and the assistance of Chip Moore, Ed Watts, and Perry Gray in post-flight analysis.

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