



MISSE PEACE Polymers Atomic Oxygen Erosion Results

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Prepared for the
2006 MISSE Post-Retrieval Conference
sponsored by the U.S. Air Force Research Laboratory
Orlando, Florida, June 26–30, 2006

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Acknowledgments

The authors would like to thank Don Jaworske of NASA GRC for coordinating all the GRC MISSE experiments. We would like to acknowledge and thank former students Jon Gummow of the Ohio Aerospace Institute, Doug Wright of Cleveland State University, and prior PEACE Team students for making and pre-flight characterizing flight and back-up samples. We thank Rachel Kamenetzky and Marshall Space Flight Center for vacuum baking flight samples. There are many companies who kindly provided samples of polymers, which we sincerely appreciate. We gratefully acknowledge Patty Hunt of Hathaway Brown School for making it possible for the students to be a part of this experiment. Finally, we would like to show our sincere appreciation to the MISSE Project Office at NASA Langley Research Center and Gary Pippin at Boeing for providing the unique opportunity to be a part of the MISSE program.

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ABSTRACT

Forty-one different polymer samples, collectively called the Polymer Erosion and Contamination Experiment (PEACE) Polymers, have been exposed to the low Earth orbit (LEO) environment on the exterior of the International Space Station (ISS) for nearly four years as part of Materials International Space Station Experiment 2 (MISSE 2). The objective of the PEACE Polymers experiment was to determine the atomic oxygen erosion yield of a wide variety of polymeric materials after long term exposure to the space environment. The polymers range from those commonly used for spacecraft applications, such as Teflon (DuPont) FEP, to more recently developed polymers, such as high temperature polyimide PMR (polymerization of monomer reactants). Additional polymers were included to explore erosion yield dependence upon chemical composition. The MISSE PEACE Polymers experiment was flown in MISSE Passive Experiment Carrier 2 (PEC 2), tray 1, on the exterior of the ISS Quest Airlock and was exposed to atomic oxygen along with solar and charged particle radiation. MISSE 2 was successfully retrieved during a space walk on July 30, 2005 during Discovery's STS-114 Return to Flight mission. Details on the specific polymers flown, flight sample fabrication, pre-flight and post-flight characterization techniques, and atomic oxygen fluence calculations are discussed along with a summary of the atomic oxygen erosion yield results. The MISSE 2 PEACE Polymers experiment is unique because it has the widest variety of polymers flown in LEO for a long duration and provides extremely valuable erosion yield data for spacecraft design purposes.

1. INTRODUCTION

Polymers such as polyimide Kapton (DuPont) and Teflon FEP (fluorinated ethylene propylene) are commonly used spacecraft materials because of their desirable properties such as flexibility, low density, and electrical, thermal and optical properties. Materials on the exterior of spacecraft are exposed to atomic oxygen (AO) in the low Earth orbit (LEO) environment. Atomic oxygen is formed when short wavelength ultraviolet radiation from the Sun photodissociates molecular oxygen in the upper atmosphere. Although AO is the predominant species in LEO (below $\approx 1,000$ km), these neutral oxygen atoms have mean free paths on the order of 10^4 m at 400 km, resulting in extremely low probabilities of re-association. As a spacecraft orbits the Earth it travels with a velocity on the order of 7.7 km/sec and rams into the oxygen atoms. The flux of AO at International Space Station (ISS) altitudes is approximately 5.23×10^{13} atoms/cm² sec for

normal incident ram surfaces (at 400 km averaged over the 11 year solar cycle), and the average energy of an oxygen atom impacting spacecraft at ram velocities is 4.5 eV [1].

A number of processes can take place when an oxygen atom strikes a spacecraft surface at orbital velocities. These include chemical reaction with surface molecules, elastic scattering, scattering with partial or full thermal accommodation, recombination, or excitation of ram species [2]. Because the final oxidation product for most polymers is a gas, AO erosion results. Materials with volatile oxidation products develop a cone-like morphology under directed AO attack. Atomic oxygen erosion of polymers in LEO is a serious threat to spacecraft performance and durability. It is therefore essential to understand the AO erosion yield, E_y , the volume loss per incident oxygen atom (cm^3/atom) of polymers for spacecraft applications.

Forty-one different polymer samples, collectively called the Polymer Erosion and Contamination Experiment (PEACE) Polymers, have been exposed to the LEO space environment on the exterior of the ISS for nearly four years as part of Materials International Space Station Experiment 1 & 2 (MISSE 1 & 2). The purpose of the MISSE PEACE Polymers experiment was to accurately determine the AO erosion yield of a wide variety of polymeric materials exposed for an extended period of time to the LEO space environment [3]. The polymers range from those commonly used for spacecraft applications, such as Teflon FEP, to more recently developed polymers, such as high temperature polyimide PMR (polymerization of monomer reactants). Additional polymers were included to explore erosion yield dependence upon chemical composition. Details on the specific polymers flown, flight sample fabrication, pre-flight and post-flight characterization techniques, and atomic oxygen fluence determination are discussed along with a summary of the atomic oxygen erosion yield results.

2. EROSION YIELD MEASUREMENTS

2.1 Erosion Yield Determination

A common technique for determining the erosion yield (E_y) of flight samples is based on mass loss and is calculated based on mass measurements before and after flight. The erosion yield of the sample is determined through the following equation:

$$E_y = \frac{\Delta M_S}{(A_S \rho_S F)} \quad (1)$$

where

E_y = erosion yield of flight sample (cm^3/atom)

ΔM_S = mass loss of the flight sample (g)

A_S = surface area of the flight sample exposed to atomic oxygen attack (cm^2)

ρ_S = density of flight sample (g/cm^3)

F = fluence of atomic oxygen (atoms/cm^2)

The AO fluence (F) can be determined through the mass loss of a Kapton H witness sample because Kapton H has a well characterized erosion yield ($3.0 \times 10^{-24} \text{ cm}^3/\text{atom}$) in the LEO environment [4]. Therefore, the AO fluence can be calculated using the following equation:

$$F = \frac{\Delta M_K}{(A_K \rho_K E_K)} \quad (2)$$

where

- F = low Earth orbit AO fluence (atoms/cm²)
 ΔM_K = mass loss of Kapton H witness sample (g)
 A_K = surface area of Kapton H witness sample exposed to atomic oxygen (cm²)
 ρ_K = density of Kapton H witness sample (1.42 g/cm³)
 E_K = erosion yield of Kapton H witness sample (3.0×10^{-24} cm³/atom)

Thus

$$E_y = E_K \frac{\Delta M_S A_K \rho_K}{\Delta M_K A_S \rho_S} \quad (3)$$

Another technique for determining erosion yield is based on recession depth measurements. Because many of the PEACE Polymers are composed of multiple thin film layers (see section 4.1) and because erosion can occur in multiple layers at one time, as shown in the Upilex-S and Kapton H samples in Section 8 below, it was decided that mass loss is the best technique for this flight experiment. In addition, individual layers can be so fragile after erosion that recession depth measurements would be impossible to obtain. With mass loss measurements, an average mass of the fragile pieces and layers and underlying non-eroded supportive layers can be obtained.

2.2 Rehydration/Dehydration Issues

One of the critical issues with using mass loss for obtaining accurate erosion yield data is that dehydrated mass measurements are needed. Many polymer materials, such as Kapton, are very hygroscopic (absorbing up to 2% of their weight in moisture) and can fluctuate in mass with humidity and temperature. Therefore, for accurate mass loss measurements to be obtained, it is necessary that the samples be fully dehydrated (i.e. in a vacuum desiccator) prior to measuring the mass for both pre-flight and post-flight.

3. PEACE POLYMERIC MATERIALS

The specific polymers chosen for the MISSE PEACE Polymers experiment represent a wide range of polymeric materials and bonding types. Included are those commonly used for spacecraft applications, such as Teflon FEP, to more recently developed polymers, such as high temperature polyimide PMR (polymerization of monomer reactants). Polymers such as polyethylene oxide and cellulose acetate are also included based solely on their chemical composition to provide LEO erosion yield data for modeling purposes. Two polyimide Kapton H samples were included to serve as fluence calibration witness samples. The 41 polymers, their associated polymer abbreviations and the MISSE serial numbers are listed in Table 1.

Table 1. MISSE PEACE Polymers List.

MISSE Serial #	Material	Abbrev.	Trade Names
2-E5-6	Acrylonitrile butadiene styrene	ABS	Cyclocac
2-E5-7	Cellulose acetate	CA	Clarifoil; Tenite Acetate; Dexel
2-E5-8	Poly-(p-phenylene terephthalamide)	PPD-T	Kevlar 29 fabric
2-E5-9	Polyethylene	PE	
2-E5-10	Polyvinyl fluoride	PVF	Tedlar TTR10SG3
2-E5-11	Crystalline polyvinylfluoride w/white pigment	PVF	White Tedlar TW10B53
2 -E5-12	Polyoxymethylene; acetal; polyformaldehyde	POM	Delrin (Natural)
2-E5-13	Polyacrylonitrile	PAN	Barex 210
2-E5-14	Allyl diglycol carbonate	ADC	CR-39, Homalite H-911
2-E5-15	Polystyrene	PS	Trycite 1000/Trycite Dew
2-E5-16	Polymethyl methacrylate	PMMA	Plexiglas; Lucite; Acrylite (Impact Mod.)
2-E5-17	Polyethylene oxide	PEO	Alkox E-30
2-E5-18	Poly(p-phenylene-2,6-benzobisoxazole)	PBO	Balanced Biaxially Film
2-E5-19	Epoxide or epoxy	EP	Hysol EA956
2-E5-20	Polypropylene	PP	Type C28
2-E5-21	Polybutylene terephthalate	PBT	GE Valox 357
2-E5-22	Polysulphone	PSU	Thermolux P1700-NT11; Udel P-1700
2-E5-23	Polyurethane	PU	Dureflex PS 8010
2-E5-24	Polyphenylene isophthalate	PPPA	Nomex Aramid Paper Type 410
2-E5-25	Graphite	PG	Pyrolytic Graphite
2-E5-26	Polyetherimide	PEI	Ultem 1000
2-E5-27	Polyamide 6 or nylon 6	PA 6	Akulon K; Ultramid B
2-E5-28	Polyamide 66 or nylon 66	PA 66	Maranyl A; Zytel
2-E5-29	Polyimide	PI	LaRC CP1 (CP1-300)
2-E5-30	Polyimide (PMDA)	PI	Kapton H
2-E5-31	Polyimide (PMDA)	PI	Kapton HN
2-E5-32	Polyimide (BPDA)	PI	Upilex-S
2-E5-33	Polyimide (PMDA)	PI	Kapton H
2-E5-34	High temperature polyimide resin	PI	PMR-15
2-E5-35	Polybenzimidazole	PBI	Celazole PBI
2-E5-36	Polycarbonate	PC	PEEREX 61 (P61)
2-E5-37	Polyetheretherketone	PEEK	Victrex PEEK 450
2-E5-38	Polyethylene terephthalate	PET	Mylar A/200
2-E5-39	Chlorotrifluoroethylene	CTFE	Neoflon CTFE M-300; Kel-F
2-E5-40	Halar ethylene-chlorotrifluoroethylene	ECTFE	Halar
2-E5-41	Tetrafluoroethylene-ethylene copolymer	ETFE	Tefzel ZM
2-E5-42	Fluorinated ethylene propylene	FEP	Teflon FEP (round robin)
2-E5-43	Polytetrafluoroethylene	PTFE	Chemfilm DF 100
2-E5-44	Perfluoroalkoxy copolymer resin	PFA	Teflon PFA CLP (200 CLP)
2-E5-45	Amorphous Fluoropolymer	AF	Teflon AF 1601
2-E5-46	Polyvinylidene fluoride	PVDF	Kynar 740

4. SAMPLE FABRICATION & PRE-FLIGHT CHARACTERIZATION

4.1 Flight Sample Dimensions & Fabrication

The MISSE PEACE Polymers samples were approximately 2.54 cm diameter disks. The polymers were most often obtained in thin film form, typically ranging from 0.0025 cm to 0.051 cm thick and were punched into the circular disks using a double bow punch cutter and an Arbor press.

Several samples did not come in film form. Allyl diglycol carbonated (ADC, 2-E5-14), typically used as lens material, was ordered from a manufacturer in 2.54 cm diameter, 0.079 cm thick samples. Pyrolytic graphite (PG, 2-E5-25) was also ordered in 2.54 cm diameter pieces, 0.203 cm thick. Hysol EA956 epoxy (EP, 2-E5-19) was purchased as a two-part kit. Flight samples were fabricated by mixing and curing the epoxy, and then carefully sawing out 2.54 cm diameter samples from brittle sheets of cured epoxy. The thickness of the flight sample is ≈ 0.231 cm thick. Polyethylene oxide (PEO, 2-E5-17) was purchased as a powder and fabricated into sheet material by pressing the powder with heated plates using a Carver Laboratory Press. To keep the PEO from sticking to the plates, two sheets of Kapton were placed in between the press plates and the PEO powder. The PEO was pressed at 23,000 lb (10.5 metric tons) for 5-10 seconds while simultaneously applying heat. The exact temperature of heating is not known, but the press heats to 260 °C (500 °F) and approximately 40% of the power was used. After pressing, when completely cooled, the Kapton sheets were carefully separated from the PEO. This resulted in a sheet of PEO between 0.74 cm and 0.94 cm thick, from which 2.54 cm diameter samples were punched out. High temperature polyimide resin, PMR-15 (PI, 2-E5-34), was fabricated by the Polymers Branch at GRC in 2.54 cm diameter, 0.030 cm thick pieces. Poly-(p-phenylene terephthalamide), also known as Kevlar 29 (PPD-T, 2-E5-8), was obtained in fabric form. Flight samples were cut out and the fabric samples were carefully wrapped in Al disks to protect the edges from fraying or losing small filaments. The extreme edge on the exposed side was covered with Al in such a way that only the Kevlar would be exposed to AO in the flight hardware. Polyphenylene isophthalate (PPPA), also known as Nomex (DuPont), was supplied as 0.005 cm thick sheets of paper, from which flight samples were easily punched out.

4.2 Sample Stacking

MISSE 1 & 2 were originally planned for a 1-year mission. The expected AO fluence for a 1-year exposure on ISS was 3.28×10^{21} atoms/cm² for directed AO exposed surfaces, based on a mission launch date of June 2001, a 400 km circular orbit and a 51.6° inclination. For many of the thin film polymers, a single layer would be completely eroded away after a 1-year mission. In addition, flight experiments are not always retrieved on the original planned date, but can be left in space much longer. Therefore, depending on the polymer thickness and estimated erosion yield, stacking of several sample layers was necessary. The total number of layers to be stacked for flight was chosen based on surviving a 3-year mission (3 times the duration of the original planned mission).

Stacking of the sample layers is complicated by the fact that increasing the mass of the sample causes a decrease in the sensitivity of the mass change. Therefore, one would ideally want to

measure the lightest sample possible before and after flight so the mass loss is a significant percent of the total mass of the sample. Keeping this in mind, two different sets of samples were stacked together for flight. The first set included the number of sample layers needed to be stacked together and weighed to survive an AO exposure period of 1.5 years. The second set was the number of additional sample layers that should be added behind the weighed set to ensure survival of a 3-year mission.

Because the MISSE experiment was planned as a 1-year mission, it was decided to choose a mission time slightly longer than expected for sample weighing (hence 1.5 years for an expected 1-year mission). Therefore, based on the expected AO fluence for a 1.5 year mission (computed as 4.55×10^{21} atoms/cm²), and an estimated erosion yield of each polymer, the number of layers for each sample needed in order to survive 1.5 years of exposure was determined. The AO fluence for a 3-year mission was computed to be 9.1×10^{21} atoms/cm² (twice the 1.5-year fluence); therefore the total number of samples to be stacked together as one “flight sample” was calculated based on surviving the 3-year fluence. This additional “stacked” (un-weighed) set was always placed behind the “weighed” set in the flight hardware. For example, the estimated erosion yield for cellulose acetate (CA) is 6.8×10^{-24} cm³/atom [5]. Based on this erosion yield and the computed AO fluence, the estimated thickness loss for 1.5 years in LEO is 0.031 cm. The sample thickness is only 0.005 cm, and therefore 7 sample layers needed to be stacked for mass measurements. A total of 0.062 cm was expected to be eroded away after 3 years, so 13 total layers needed to be stacked for flight (the 7 weighed samples + 6 additional samples stacked behind). In all, a total of 205 individual sample layers were flown.

For each complete flight sample, an identical back-up sample was prepared and characterized. All individual sample layers were carefully scribe marked at the edges indicating sample ID, orientation (front), flight or back-up sample and whether the sample had been vacuum heat treated.

4.3 Outgassing & Vacuum Heat Treatment

Samples to be flown in the space environment need to meet outgas requirements as outlined in ASTM E 595, the Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment [6]. This test method evaluates, under carefully controlled conditions, the changes in the mass of a test specimen on exposure under vacuum to a temperature of 125 °C (total mass loss (TML)), and the mass of those products that leave the specimen and condense on a collector plate at a temperature of 25 °C (collected volatile condensable materials (CVCM)) [6]. MISSE management decided that material being flown as part of MISSE should meet the historical screening levels of <1.00% TML and <0.10% CVCM. Ten of the 41 PEACE polymers did not meet the outgas requirements either based on previous testing or because they had not been tested, and it was decided that these polymers could be flown if they were vacuum heat treated prior to flight to remove volatile products. Vacuum heat treatment was conducted at the NASA Marshall Space Flight Center (MSFC) at a pressure of 5×10^{-7} torr (7×10^{-5} Pa). Ideally, samples were to be vacuum baked for 24 hours at 125 °C (similar to ASTM E 595), but some polymers were heated at lower temperatures due to low maximum operating temperatures. Table 2 lists the vacuum heat treated

samples, their maximum operating temperatures and the vacuum heat treatment temperature and time.

Table 2. List of Vacuum Heat Treated MISSE PEACE Polymers.

MISSE Serial #	Material	Abbreviation/ Trade Name	Max. Operating Temperature (°C)	Vacuum Heat Treatment
2-E5-6	Acrylonitrile butadiene styrene	ABS/Cycolac	105	24 hr @ 90 °C
2-E5-7	Cellulose acetate	CA/Clarifoil	230-245	68.75 hr @ 128 °C
2-E5-8	Poly-(p-phenylene terephthalamide) fabric in Al foil	PPD-T/Kevlar fabric	149-177	24 hr @ 128 °C
2-E5-10	Polyvinyl fluoride	PVF/Tedlar	107	36 hr @ 100 °C
2-E5-13	Polyacrylonitrile	PAN/Barex	200-210	24.25 hr @ 126 °C
2-E5-17	Polyethylene oxide	PEO/Alkox	65	24 hr @ 60 °C
2-E5-24	Polyphenylene isophthalate	PPPA/Nomex paper	220	24 hr @ 125 °C
2-E5-27	Polyamide 6 or nylon 6	PA 6/Akulon	98	24 hr @ 90 °C
2-E5-28	Polyamide 66 or nylon 66	PA 66/Maranyl	120	24 hr @ 90 °C
2-E5-35	Polybenzimidazole	PBI/Celazole	343	24 hr @ 125 °C

The polymer PBI (2-E5-35) curled severely at its edges during vacuum heat treatment. For this polymer, 4 individual sample layers needed to be stacked in order to survive a 3-year exposure. Therefore, the 4 layers were stacked together (after mass measurements were obtained) and held flat by mounting them in an aluminum holder for flight (similar to the one used for the Kevlar fabric).

4.4 Pre-flight Dehydrated Mass Measurements

All pre-flight mass measurements were obtained for dehydrated sample sets and after vacuum heat treatment were applicable. All sample sets were dehydrated in a vacuum desiccator maintained at a pressure of 60-100 mtorr with a mechanical roughing pump. Typically 5 flight sample sets and their corresponding back-up sets were placed together in a desiccator (a total of 10 groups of samples). The sets of samples were placed in the desiccator in a particular order and left under vacuum for a minimum of 4 days. The exact time that each sample was under vacuum was recorded. In order to maintain each sample under vacuum while weighing other samples, the vacuum desiccator would be put back under vacuum immediately after an individual sample was removed. The time at which the sample was removed from the desiccator was recorded along with the times at which it was weighed (a total of 3 readings were obtained and averaged). Previous tests showed that the mass of a dehydrated sample was not adversely affected if the desiccator was opened and quickly closed again and pumped back down to approximately 150 mtorr prior to that sample being weighed. This process allows multiple samples to be dehydrated together. The samples were weighed using either a Mettler Balance 3M (0.001 mg) or a Sartorius Balance R160P (0.00001 g), depending on their total mass. Records of the following were kept: the sequence of sample weighing, the number of samples in each set, the time under vacuum prior to weighing, the temperature and humidity in the room, the time a sample was taken out of the desiccator, the time of each weighing and the mass. The same procedure and sequence was repeated with the same samples post-flight.

4.5 Density Data

Density measurements were obtained for 36 of the 41 polymers using calibrated density gradient columns. The density solvents for the majority of polymers were made from cesium chloride (CsCl), which has a density of $\approx 2 \text{ g/cm}^3$, and water (H₂O), which has a density of 1 g/cm^3 . A more dense solution and a less dense solution were mixed from the solvents, and the more dense solution was slowly added to a 50 mL buret with increasing amounts of the less dense solution until only the less dense solution was being added, creating the density gradient. Because of the variation of density in the polymers, columns with variations in the initial mixed solutions were made to accommodate a wider range of densities to be determined. Density standards of known densities ($\pm 0.0001 \text{ g/cm}^3$) were placed in the column. Data was obtained by placing very small pieces of the polymers into the column and noting their respective positions. The sample densities were then obtained from a curve fitted from the standards. As one example, for a density column with a density range of 1.4-1.6 g/cm^3 , the more dense solution was made with a density of 1.65 g/cm^3 (13 mL CsCl & 7 mL of H₂O) and the less dense solution was made with a density of 1.38 g/cm^3 (38 mL CsCl & 62 mL of H₂O). The density solvents for the higher density polymers, such as the fluoropolymers, were carbon tetrachloride (CCl₄), which has a density of 1.594 g/cm^3 , and bromoform (CHBr₃), which has a density of 2.899 g/cm^3 . The densities for polymers either out of the range of the density columns or of a form not easily sectioned for density column measurements were obtained from either referenced literature and/or manufacturers' Materials Safety and Data Sheets.

4.6 Area Measurements

The exposed sample area was determined by measuring the diameter of the sample tray opening using digital calipers. Each specific tray opening was measured at 10 different positions to determine an average diameter. This diameter was then used to compute the sample area exposed to LEO AO for each sample position.

5. FLIGHT SAMPLE MOUNTING (TRAY E5)

The MISSE PEACE Polymers resided in sample tray E5, which holds a total of 46 1-in.-diameter flight samples. The PEACE Polymer samples start in the top row, 6th sample position from the left (i.e. ABS, with serial number 2-E5-6, represents a sample in MISSE Passive Experiment Container (PEC) 2, tray E5, sample position 6). Five other samples (4 DC 93-500 silicone samples and 1 AO scattering chamber) were also on this tray, in positions 2-E5-1 through 2-E5-5. Sample positions were chosen based on their anticipated erosion yields or by grouping samples by polymer family (such as the polyimides). High erosion yield samples were located next to the silicone samples as the high erosion yield samples are less likely to be affected by silicone cross-contamination. Fig. 1 shows a pre-flight photo of the 41 MISSE PEACE Polymers samples in tray E5 along with the 5 other samples.



Fig. 1. GRC's 41 MISSE PEACE Polymers loaded into sample tray E5. Samples with circles (2-E5-12 through 2-E5-17) were expected to have high erosion yields, samples within the solid line block are from the polyimide family and samples within the dashed line block are from the fluoropolymer family.

6. MATERIALS INTERNATIONAL SPACE STATION EXPERIMENT (MISSE)

Managed by Langley Research Center (LaRC), MISSE is a collaborative effort among NASA centers, the U.S. Air Force and private industry [7]. MISSE consists of suitcase-like experiment trays exposed to either AO and solar radiation, or solar radiation with minimal AO exposure, along with charged particle radiation and vacuum on the exterior of ISS. The PEACE Polymers were located on PEC 2 tray 1, exposed to AO along with solar and charge particle radiation. MISSE 1 & 2 were transported to the ISS, and MISSE 2 was attached to the exterior Quest Airlock on August 16, 2001 during the STS-105 shuttle mission. Fig. 2 is a photograph of MISSE PEC 2 on the ISS Quest Airlock just after attachment. The MISSE 2 PEACE Polymers tray is visible in this photograph. Fig. 3 is a photograph of the Quest Airlock showing the location of MISSE PEC 2. MISSE 1 & 2 were the first exterior experiments on the ISS hull. Although originally planned as a 1-year mission, retrieval of MISSE 1 & 2 was significantly delayed due to the Columbia Shuttle accident. MISSE 1 & 2 were successfully retrieved during a space walk on July 30, 2005 during Discovery's STS-114 Return to Flight mission after nearly 4 years of space exposure.

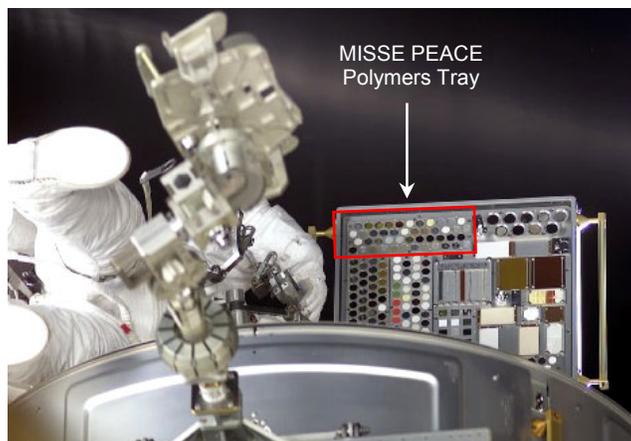


Fig. 2. During a space walk on August 16, 2001, astronaut Patrick Forrester installs MISSE PEC 2 on the ISS Quest Airlock [NASA photo STS105E5302].

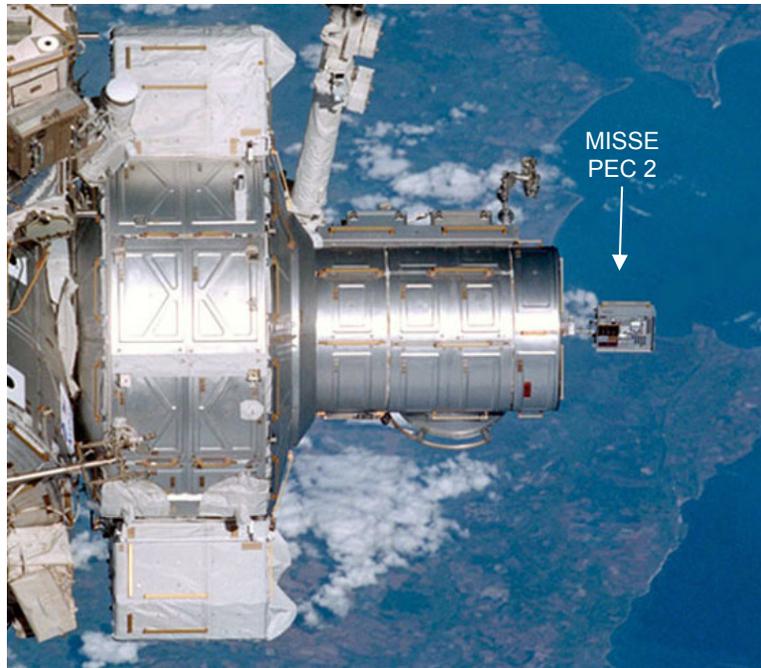


Fig. 3. Photograph of the Quest Airlock and MISSE PEC 2 taken during the STS-105 mission [NASA photo STS105-329-028].

7. MISSE 2 ENVIRONMENTAL EXPOSURE

The atomic oxygen fluence exposure for the MISSE 2 PEACE Polymers is described in Section 8 below. Estimated environmental conditions of solar exposure, tray temperatures, and ionizing radiation doses on MISSE 1 & 2 are described in detail by Pippin [8]. For PEC 2 tray (exposed to AO and solar radiation) the total equivalent sun hours (ESH) was estimated to be 5,000 – 6,700 ESH. This total includes Earth-reflected illumination (650-820 ESH). The spectrum of Earth reflection solar radiation has a reduced contribution from the vacuum ultraviolet (UV) portion of the spectrum due to absorption by the Earth's atmosphere [8]. Tray E5 was computed to have received approximately 6,300 ESH. Pippin also describes the base plate thermal cycling temperature range for MISSE 1 & 2 to be nominally between +40 °C and -30 °C with occasional short-term excursions to more extreme temperatures. The almost 4-year duration resulted in approximately 22,800 thermal cycles. Preliminary data from [8] also provided insight into the ionizing radiation environment on MISSE 1 & 2. Data from thermo-luminescent dosimeters (TLDs) with thin shielding layers are most relevant to estimates of doses for the PEACE Polymer experiment thin polymer films. The TLD data indicated receiving approximately 16 krad(Si) through 0.005 cm aluminum for MISSE 2 [8].

Black light inspection of the trays showed minimal to no contamination on the MISSE surfaces [8]. Results of x-ray photoelectron spectroscopy (XPS) contamination analysis of two MISSE 2 sapphire witness samples in tray E6 (located next to tray E5) indicated an extremely thin silica contaminant layer (1.3 and 1.4 nm on each slide, respectively) [9]. A small amount of fluorine was also detected [9].

8. MISSE 2 PEACE POLYMERS POST-FLIGHT CHARACTERIZATION

Fig. 4 is a post-retrieval photograph of the PEACE Polymers experiment tray as observed directly after PEC 2 was opened for the first time in a NASA Langley Research Center clean room. As expected, many of the polymer samples were significantly degraded, and the process of eroding through the various sample layers was evident. Fig. 5 is a post-flight photograph of the PEACE Polymers experiment (tray E5) after de-integration from MISSE PEC 2.



Fig. 4. Photograph of the PEACE Polymers experiment tray taken in the NASA Langley Research Center clean room during post-flight retrieval examination.



Fig. 5. Post-flight photograph of the MISSE 2 PEACE Polymers experiment tray.

Fortunately, even though this experiment received nearly 4 years of atomic oxygen exposure, material was left for all samples except for one (PBI, 2-E5-35). Therefore, planning for a 3 year exposure (vs. 1 yr) was found to be crucial to the experiment success.

Once tray E5 was brought back to NASA Glenn Research Center, post-flight photo-documentation of the samples was conducted. Photos were taken of the individual samples in tray E5 after removal from the tray (left in a flight stack next to the back-up stack), and also with the individual sample layers spread out. Details on the number of sample layers eroded through, those textured, and the number remaining non-eroded were recorded. When additional stacked layer sets had been placed behind the weighed sample set as a flight sample, those two groups of sample sets were separated for weighing as individual sets. Post-flight, an ideal situation would be where only the weighed set had erosion, so that the un-weighed stacked layers do not need to be weighed post-flight for erosion yield determination. Occasionally, the individual layers could not be separated from each other without damaging the sample texture or losing gossamer pieces of residual material. In these cases all layers were kept together for post-flight sample weighing.

For post-flight mass measurements, there were three situations: 1). Either a stacked set of sample layers was flown behind the weighed set with erosion only occurring within the weighed set (hence post-flight the stacked set did not need to be weighed), or only a single sample layer was flown. These ideal situations are referred to as the “Single” situation. 2). Both a weighed set and a stacked set were flown with erosion occurring through the weighed set down into the stacked set, and the two sets of layers could be separated and weighed individually post-flight (this is referred to as the “Separate” situation). Or, 3). Both a weighed set and a stacked set were flown with erosion occurring through the weighed set down into the stacked set, and the two sets of layers could not be separated post-flight without damaging the gossamer sample layers, and therefore the sample sets were weighed together post-flight (this is referred to as the “Together” situation).

The post-flight mass procedures replicated the pre-flight mass procedures in terms of the vacuum dehydration process (dehydration time, sample order, weighing technique) as often as possible.

Figs. 6, 7 and 8 provide examples of the “Single” situation. In Fig. 6 a single thick (0.203 cm) layer of pyrolytic graphite (PG, 2-E5-25) was flown and the flight sample is shown post-flight next to the non-flown control sample. Mass loss was therefore computed simply from the dehydrated mass prior to, and after flight, of this single layer.

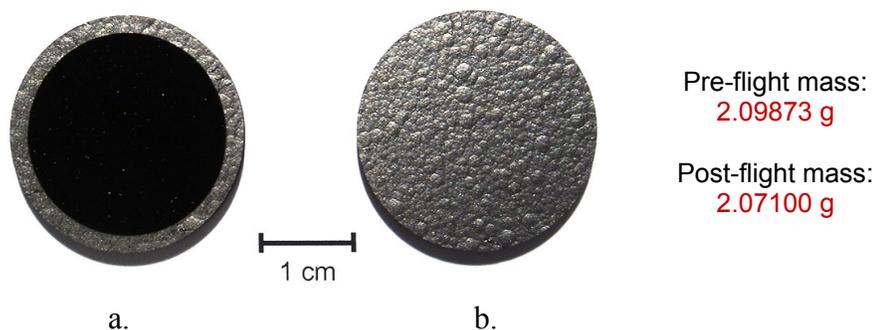


Fig. 6. Post-flight photograph of pyrolytic graphite (PG) flight and control samples (1 layer 80 mil (2,032 μ m) thick): a). Flight sample (2-E5-25), and b). Back-up sample.

The Upilex-S sample (2-E5-32) provides another example of the “Single” situation. Fig. 7 shows a post-flight photograph of the stacked flight sample next to a stacked back-up (control)

sample. A total of 11 layers were stacked together and flown, and the individual layers are shown in Figure 8 along with the back-up sample. Pre-flight, the top six of 11 layers were weighed. During flight only the top 4 layers were eroded (the top 2 completely eroded leaving the E5 tray-protected ring, the 3rd layer was eroded with holes at the AO textured cone valleys, and the 4th layer was eroded at the cone valley hole locations in the 3rd layer). Therefore, post-flight, the top 6 layers were separated from the bottom 5 layers and only the top set was weighed and directly compared to the mass of the top 6 layers pre-flight for flight experiment mass loss.



Fig. 7. Post-flight photograph of *Upilex-S* flight and control samples (11 layers each 0.0025 cm thick): a). Flight sample stack (2-E5-32), and b). Back-up sample stack.

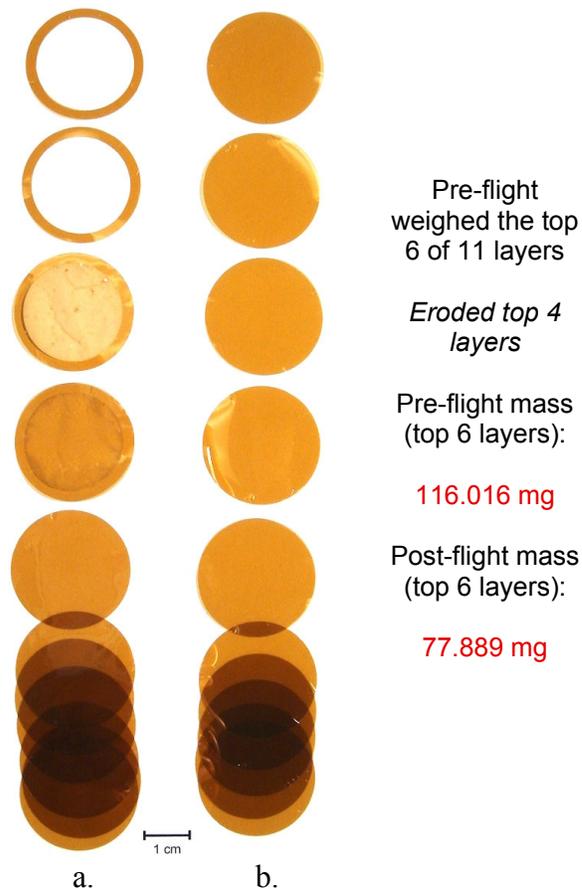


Fig. 8. Post-flight photograph of *Upilex-S* flight and back-up samples (11 layers each 0.025 cm thick): a). Flight sample (2-E5-32), and b). Back-up sample.

Atomic oxygen fluence witness sample Kapton H (2-E5-30) provides an example of a “Separate” situation. As shown in Fig. 9, three 0.0127 cm thick layers were flown. Prior to flight only the top 2 layers were weighed. During flight, AO eroded the top layer completely and eroded the 2nd layer resulting in the development of the cone-like texture. In the bottom of the valleys between the cones there were holes providing exposure to the sample layer below. Even though the 2nd layer was still intact (and could be weighed as a separate layer), the 3rd layer was also textured. Therefore, in this case, pre-flight and post-flight mass measurements are needed for both the weighed top 2 layers and the bottom “un-weighed” layer.

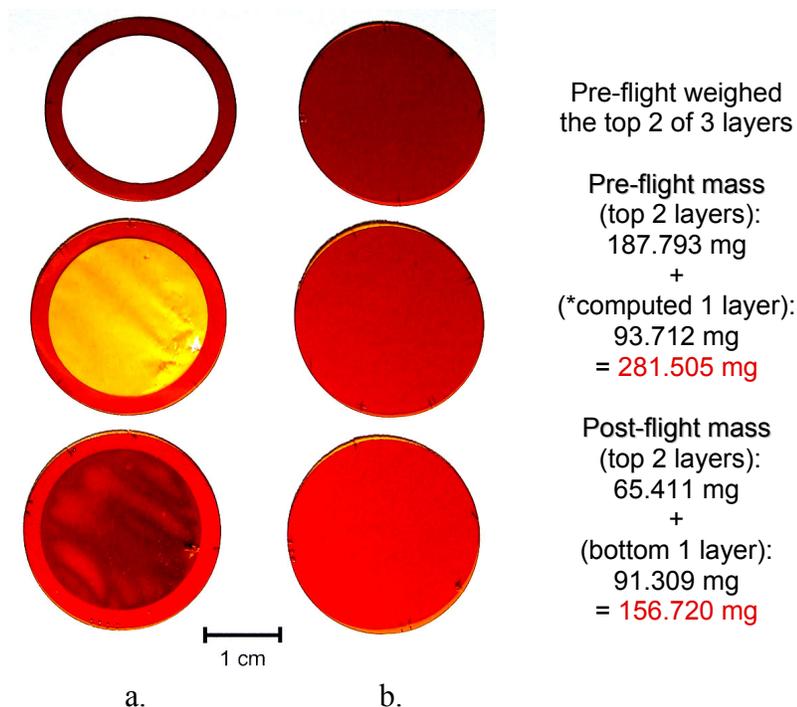


Fig. 9. Post-flight photograph of Kapton H flight and back-up samples (3 layers each 0.0127 cm thick): a). Flight sample (2-E5-30), and b). Back-up sample.

Fig. 10 provides a schematic diagram of a situation where a stack of un-weighed layers has been flown behind a weighed layer set and during flight AO has eroded through the weighed set into the un-weighed set. In this situation, the pre-flight mass of the stacked set (M_S) needed to be computed. This mass was computed based on taking an average of both the weighed flight set (M_F) and the weighed back-up sample set (M_B) prior to flight to get an individual layer average and then multiplying that mass by the number of layers (n) in the un-weighed stack set. This equation, along with the variables, is shown in Fig. 10.

9. MISSE 2 TRAY 1 E5 AO FLUENCE

The AO fluence for PEC 2 tray 1 E5 was computed based on the mass loss of two Kapton H witness samples (2-E5-30 & 2-E5-33). Each Kapton H witness sample consisted of three 127 μm thick layers for a total thickness of 381 μm . As stated previously, Kapton H is commonly used for fluence calibration because it has a well-characterized erosion yield in LEO (3.0×10^{-24} atoms/ cm^2) [4]. The AO fluence was determined using vacuum-dehydrated mass loss based on 2

techniques: 1). Averaging the 1st three mass readings, and 2). Back-extrapolation of the mass data to time zero using linear curve fits to get theoretically dehydrated mass values. The results of these measurements are provided in Table 3. The AO fluence values for the individual samples using the same techniques were very similar. The average AO fluences for the two techniques were also very similar. Therefore, it was decided to use the average mass value instead of the linear back-extrapolation value for determining both the LEO AO fluence for the experiment and for determining the erosion yield values for the individual PEACE Polymer samples. The average AO fluence for the PEACE Polymers experiment exposure (tray E5) on MISSE 2, based on the two Kapton H witness samples (averaging the 1st three mass readings), was determined to be 8.43×10^{21} atoms/cm².

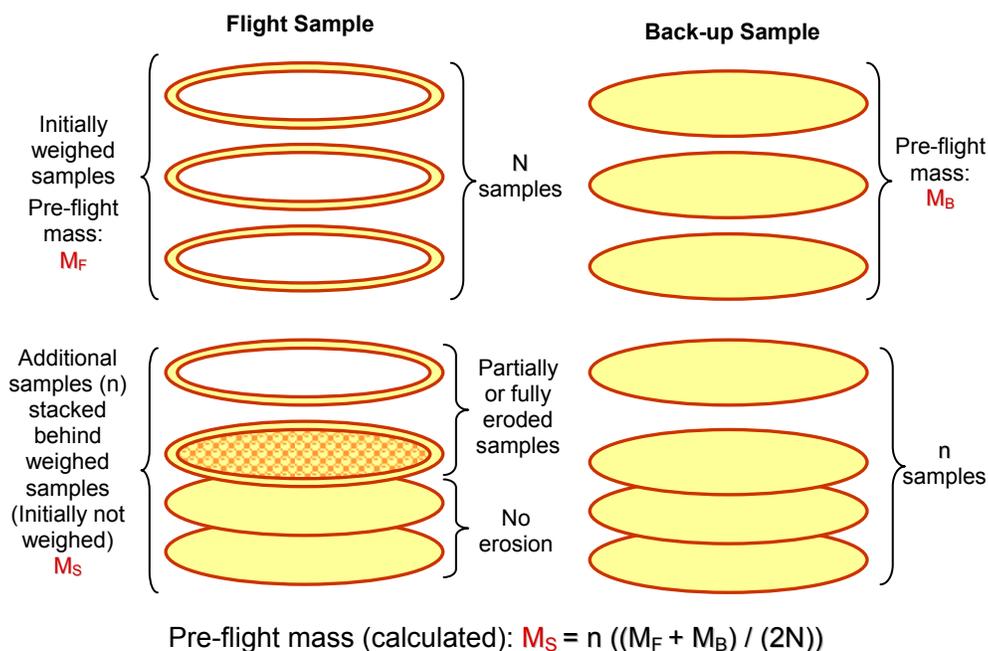


Fig. 10. Diagram describing calculation for the pre-flight mass of the un-weighed stacked flight layers.

Table 3. AO Fluence for Kapton H Calibration Samples.

Mass Loss Technique	Kapton H 2-E5-30 Mass Loss (g)	Kapton H 2-E5-30 AO Fluence (atoms/cm ²)	Kapton H 2-E5-33 Mass Loss (g)	Kapton H 2-E5-33 AO Fluence (atoms/cm ²)	Average AO Fluence (atoms/cm ²)
Ave. of 1 st 3 readings	0.124785	8.425 E+21	0.129219	8.436 E+21	8.43 E+21
Back extrapolation to time zero	0.124579	8.411 E+21	0.128811	8.409 E+21	8.41 E+21

10. LEO EROSION YIELD DATA

Table 4 provides the erosion yield data for the MISSE 2 PEACE Polymers experiment along with the sample mass loss, density and exposure area. Six of the 41 samples were either fully or partially eroded through (such as at one edge) after the 4 year mission. Therefore, the erosion

yield is actually greater than the value provided, as indicated in Table 4. These samples include the following:

- 2-E5-9 PE (6 layers, 0.005 cm thick) eroded with residual material left
- 2-E5-14 ADC (1 layer, 0.079 cm thick) eroded at 1 edge, sample curled up
- 2-E5-16 PMMA (10 layers, 0.005 cm thick) eroded through in center, residual material left
- 2-E5-26 PEI (2 layers, 0.025 cm thick) textured back surface (AO valleys eroded through)
- 2-E5-34 PMR-15 (1 layer, 0.30 cm thick) eroded through at 1 edge
- 2-E5-35 PBI (14 layers, 0.005 cm thick) all 4 layers completely eroded away

11. FUTURE WORK

Error analyses will be conducted to determine the error in the erosion yield values for each of the MISSE 2 PEACE Polymers flight samples. A handbook will be published with specific details on each of the individual flight samples. The MISSE 2 PEACE Polymers erosion yield values will eventually be compared with erosion yield values from the MISSE 5 PEACE Polymers experiment, currently being exposed to atomic oxygen with minimum solar radiation on the exterior of ISS, so that synergistic effects of solar exposure on AO erosion yield can be determined for long duration exposures.

12. SUMMARY & CONCLUSIONS

Forty-one different polymer samples, collectively called the PEACE Polymers, have been exposed to the LEO space environment on the exterior of the ISS for nearly four years as part of MISSE 2. The objective of this experiment was to accurately determine the atomic oxygen erosion yield of a wide variety of polymeric materials after long term exposure to the space environment. The MISSE 2 PEACE Polymers experiment was exposed to atomic oxygen along with solar and charged particle radiation. Mass loss was determined to be the best technique for characterizing the atomic oxygen erosion yield values for this experiment because erosion sometimes occurred in underlying layers at “cone valleys” in partially eroded upper layers, and because some layers were fragile gossamer films.

Atomic oxygen erosion yield values were determined for the MISSE 2 PEACE Polymers experiment based on mass loss obtained from pre-flight and post-flight vacuum dehydrated samples. Exposure areas were determined from individual flight sample hardware openings. Density values for 36 of the 41 polymers were obtained using calibrated density gradient columns. The AO fluence for the experiment was determined based on two Kapton H witness samples. Back-extrapolation of the mass data to time zero (to get a theoretically dehydrated mass) as compared to using the average mass values indicated no significant difference in AO fluence between these techniques. The average atomic oxygen fluence for the MISSE 2 PEACE Polymers experiment (MISSE 2, tray 1, sample tray E5) was determined to be 8.43×10^{21} atoms/cm² based on average mass values. There was partial or complete erosion of 6 of the 41 flight samples, and hence the erosion yield values for these samples are greater than the value reported. Planning for a 3 year exposure (vs. 1 yr) was found to be crucial to the success of this experiment.

The MISSE 2 PEACE Polymers experiment is unique because it has the widest variety of polymers flown in LEO for a long duration under identical conditions and provides extremely valuable erosion yield data for spacecraft design and predictive model development purposes.

Table 4. MISSE 2 PEACE Polymers Erosion Yield Data.

MISSE Serial #	Material	Polymer Abbrev.	MISSE 2 Mass Loss (g)	Density (g/cm ³)	Area (cm ²)	MISSE 2 Erosion Yield (cm ³ /atom)
2-E5-6	Acrylonitrile butadiene styrene	ABS	0.033861	1.0500	3.4944	1.09E-24
2-E5-7	Cellulose acetate	CA	0.191482	1.2911	3.4831	5.05E-24
2-E5-8	Poly-(p-phenylene terephthalamide)	PPD-T (Kevlar)	0.026790	1.4422	3.5099	6.28E-25
2-E5-9	Polyethylene	PE	0.102760	0.9180	3.5489	> 3.74E-24
2-E5-10	Polyvinyl fluoride	PVF (Tedlar)	0.132537	1.3792	3.5737	3.19E-24
2-E5-11	Crystalline polyvinylfluoride w/white pigment	PVF (White Tedlar)	0.004714	1.6241	3.4176	1.01E-25
2-E5-12	Polyoxymethylene; acetal; polyformaldehyde	POM (Delrin)	0.378378	1.3984	3.5119	9.14E-24
2-E5-13	Polyacrylonitrile	PAN	0.047281	1.1435	3.4768	1.41E-24
2-E5-14	Allyl diglycol carbonate	ADC (CR-39)	0.267295	1.3173	3.5392	> 6.80E-24
2-E5-15	Polystyrene	PS	0.115947	1.0503	3.5043	3.74E-24
2-E5-16	Polymethyl methacrylate	PMMA	0.194588	1.1628	3.5456	> 5.60E-24
2-E5-17	Polyethylene oxide	PEO	0.066395	1.1470	3.5591	1.93E-24
2-E5-18	Poly(p-phenylene-2,6-benzobisoxazole)	PBO (Zylon)	0.056778	1.3976	3.5526	1.36E-24
2-E5-19	Epoxide or epoxy	EP	0.140720	1.1150	3.5576	4.21E-24
2-E5-20	Polypropylene	PP	0.072357	0.9065	3.5336	2.68E-24
2-E5-21	Polybutylene terephthalate	PBT	0.036429	1.3318	3.5619	9.11E-25
2-E5-22	Polysulphone	PSU	0.105948	1.2199	3.5010	2.94E-24
2-E5-23	Polyurethane	PU	0.057227	1.2345	3.5182	1.56E-24
2-E5-24	Polyphenylene isophthalate	PPPA (Nomex)	0.030549	0.7200	3.5626	1.41E-24
2-E5-25	Graphite	PG	0.02773	2.2200	3.5703	4.15E-25
2-E5-26	Polyetherimide	PEI	0.126853	1.2873	3.5352	> 3.31E-24
2-E5-27	Polyamide 6 or nylon 6	PA 6	0.118376	1.1233	3.5646	3.51E-24
2-E5-28	Polyamide 66 or nylon 66	PA 66	0.065562	1.2252	3.5249	1.80E-24
2-E5-29	Polyimide	PI (CP1)	0.080648	1.4193	3.5316	1.91E-24
2-E5-30	Polyimide (PMDA)	PI (Kapton H)	0.124780	1.4273	3.4590	3.00E-24
2-E5-31	Polyimide (PMDA)	PI (Kapton HN)	0.121315	1.4345	3.5676	2.81E-24
2-E5-32	Polyimide (BPDA)	PI (Upilex-S)	0.038127	1.3866	3.5382	9.22E-25
2-E5-33	Polyimide (PMDA)	PI (Kapton H)	0.129250	1.4273	3.5773	3.00E-24
2-E5-34	High temperature polyimide resin	PI (PMR-15)	0.118887	1.3232	3.5256	> 3.02E-24
2-E5-35	Polybenzimidazole	PBI	0.082708	1.2758	3.4762	> 2.21E-24
2-E5-36	Polycarbonate	PC	0.142287	1.1231	3.5010	4.29E-24
2-E5-37	Polyetheretherkeytone	PEEK	0.107764	1.2259	3.4821	2.99E-24
2-E5-38	Polyethylene terephthalate	PET (Mylar)	0.125187	1.3925	3.5432	3.01E-24
2-E5-39	Chlorotrifluoroethylene	CTFE (Kel-f)	0.052949	2.1327	3.5452	8.31E-25
2-E5-40	Halar ethylene-chlorotrifluoroethylene	ECTFE (Halar)	0.088869	1.6761	3.5103	1.79E-24
2-E5-41	Tetrafluoroethylene-ethylene copolymer	ETFE (Tefzel)	0.049108	1.7397	3.4854	9.61E-25
2-E5-42	Fluorinated ethylene propylene	FEP	0.012479	2.1443	3.4468	2.00E-25
2-E5-43	Polytetrafluoroethylene	PTFE	0.008938	2.1503	3.4841	1.42E-25
2-E5-44	Perfluoroalkoxy copolymer resin	PFA	0.010785	2.1383	3.4570	1.73E-25
2-E5-45	Amorphous Fluoropolymer	AF	0.012352	2.1463	3.4544	1.98E-25
2-E5-46	Polyvinylidene fluoride	PVDF (Kynar)	0.066860	1.7623	3.4993	1.29E-24

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (<i>Leave blank</i>)		2. REPORT DATE November 2006	3. REPORT TYPE AND DATES COVERED Technical Memorandum	
4. TITLE AND SUBTITLE MISSE PEACE Polymers Atomic Oxygen Erosion Results			5. FUNDING NUMBERS WBS 698671.01.03.36	
6. AUTHOR(S) Kim K. de Groh, Bruce A. Banks, Catherine E. McCarthy, Rochelle N. Rucker, Lily M. Roberts, and Lauren A. Berger				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-15774	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2006-214482	
11. SUPPLEMENTARY NOTES Prepared for the 2006 MISSE Post-Retrieval Conference sponsored by the U.S. Air Force Research Laboratory, Orlando, Florida, June 26-30, 2006. Kim K. de Groh and Bruce A. Banks, NASA Glenn Research Center; Catherine E. McCarthy, Rochelle N. Rucker, Lily M. Roberts, and Lauren A. Berger, Hathaway Brown School, 19600 North Park Blvd., Shaker Heights, Ohio 44122. Responsible person, Kim K. de Groh, organization code RP4, 216-433-2297.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Categories: 18 and 27 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (<i>Maximum 200 words</i>) Forty-one different polymer samples, collectively called the Polymer Erosion and Contamination Experiment (PEACE) Polymers, have been exposed to the low Earth orbit (LEO) environment on the exterior of the International Space Station (ISS) for nearly 4 years as part of Materials International Space Station Experiment 2 (MISSE 2). The objective of the PEACE Polymers experiment was to determine the atomic oxygen erosion yield of a wide variety of polymeric materials after long term exposure to the space environment. The polymers range from those commonly used for spacecraft applications, such as Teflon (DuPont) FEP, to more recently developed polymers, such as high temperature polyimide PMR (polymerization of monomer reactants). Additional polymers were included to explore erosion yield dependence upon chemical composition. The MISSE PEACE Polymers experiment was flown in MISSE Passive Experiment Carrier 2 (PEC 2), tray 1, on the exterior of the ISS Quest Airlock and was exposed to atomic oxygen along with solar and charged particle radiation. MISSE 2 was successfully retrieved during a space walk on July 30, 2005, during Discovery's STS-114 Return to Flight mission. Details on the specific polymers flown, flight sample fabrication, pre-flight and post-flight characterization techniques, and atomic oxygen fluence calculations are discussed along with a summary of the atomic oxygen erosion yield results. The MISSE 2 PEACE Polymers experiment is unique because it has the widest variety of polymers flown in LEO for a long duration and provides extremely valuable erosion yield data for spacecraft design purposes.				
14. SUBJECT TERMS Atomic oxygen; International Space Station; Space flight experiment; Environmental degradation; Erosion yield; Polymers			15. NUMBER OF PAGES 24	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

