



Embrittlement of MISSE 5 Polymers After 13 Months of Space Exposure

*Aobo Guo, Grace T. Yi, Claire C. Ashmead, and Gianna G. Mitchell
Hathaway Brown School, Shaker Heights, Ohio*

*Kim K. de Groh
Glenn Research Center, Cleveland, Ohio*

*Bruce A. Banks
Alphaport, Inc., Cleveland, Ohio*

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National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

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Hathaway Brown School
Shaker Heights, Ohio 44122

Kim K. de Groh
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Bruce A. Banks
Alphaport, Inc.
Cleveland, Ohio 44135

Abstract

Understanding space environment induced degradation of spacecraft materials is essential when designing durable and stable spacecraft components. As a result of space radiation, debris impacts, atomic oxygen interaction, and thermal cycling, the outer surfaces of space materials degrade when exposed to low Earth orbit (LEO). The objective of this study was to measure the embrittlement of 37 thin film polymers after LEO space exposure. The polymers were flown aboard the International Space Station and exposed to the LEO space environment as part of the Materials International Space Station Experiment 5 (MISSE 5). The samples were flown in a nadir-facing position for 13 months and were exposed to thermal cycling along with low doses of atomic oxygen, direct solar radiation and omni-directional charged particle radiation. The samples were analyzed for space-induced embrittlement using a bend-test procedure in which the strain necessary to induce surface cracking was determined. Bend-testing was conducted using successively smaller mandrels to apply a surface strain to samples placed on a semi-suspended pliable platform. A pristine sample was also tested for each flight sample. Eighteen of the 37 flight samples experienced some degree of surface cracking during bend-testing, while none of the pristine samples experienced any degree of cracking. The results indicate that 49 percent of the MISSE 5 thin film polymers became embrittled in the space environment even though they were exposed to low doses (~2.75 krad (Si) dose through 127 μm Kapton) of ionizing radiation.

Introduction

As a result of space radiation, debris impacts, atomic oxygen interaction, and thermal cycling, the outer surfaces of space materials degrade when exposed to low Earth orbit (LEO) (Ref. 1). As ionizing radiation is known to embrittle polymers and has severely embrittled the Teflon fluorinated ethylene propylene (FEP) outer layer of the multilayer insulation covering the Hubble Space Telescope (Ref. 2). A study was conducted to measure the embrittlement of 37 thin film polymers after LEO space exposure. The polymers were flown aboard the International Space Station and exposed to the LEO space environment for 13 months as part of the Materials International Space Station Experiment 5 (MISSE 5).

MISSE is a series of spaceflight experiments designed to test the performance and durability of materials and devices exposed to the low Earth orbit (LEO) space environment. MISSE consists of 11 flight experiment trays (10 Passive Experiment Carriers (PECs) and one smaller tray being flown with MISSE 8) that are mounted to the exterior of the International Space Station (ISS). Each two-sided tray contains numerous individual flight experiments and are positioned in either a ram/wake orientation or a zenith/nadir orientation (Ref. 3). The MISSE 5 polymer samples were from the Polymer Erosion and Contamination Experiment (PEACE) and were flown in a nadir-facing position for 13 months, which resulted in exposure to omni-directional charged particle radiation, thermal cycling, and low doses of atomic oxygen and direct solar radiation (Ref. 3). The samples were analyzed for space-induced embrittlement using a bend-test procedure in which the strain necessary to induce surface cracking was determined. Details on the MISSE 5 experiment, polymer flight samples, test procedures, and bend-test results are provided.

MISSE 5 Experiments and Exposure

MISSE 5 contained two active and one passive experiments: The Forward Technology Solar Cell Experiment (FTSCE), an active experiment that tested the performance of 36 current and advanced generation solar cells for use on future spacecraft; the active Second Prototype Communication Satellite System (PCSat-2) that provided a communications system and tested the Amateur Satellite Service off-the-shelf solution for telemetry command and control; and the passive MISSE 5 Thermal Blanket Materials Experiment, which consisted of several individual experiments to measure the degradation of more than 200 materials in the space environment (Ref. 3). The 37 PEACE samples were flown as part of the MISSE 5 Thermal Blanket Materials Experiment.

MISSE 5 was placed in a zenith/nadir position on the P6 Trunion Pin Handrail of the International Space Station (ISS) during the STS-114 mission on August 3, 2005. Figure 1 shows a pre-flight photograph of MISSE 5 and an on-orbit photo taken during the STS-114 mission. MISSE 5 was exposed to the LEO space environment for 13 months, and was retrieved on September 15, 2006 during the STS-115 mission.

It is estimated that the polymers received an ionizing radiation (also called charged particle radiation) dose of ~ 2.75 krad(Si) through $127 \mu\text{m}$ Kapton, 165 ± 25 direct Sun hours (~ 360 Earth reflected), an atomic oxygen fluence of $\sim 1.8 \times 10^{20}$ atoms/cm², and 6400 thermal cycles (Ref. 4). Temperature range was estimated from the experiment deck temperature for the Forward Technology Solar Cell Experiment on the solar facing side of MISSE 5 (Ref. 5).

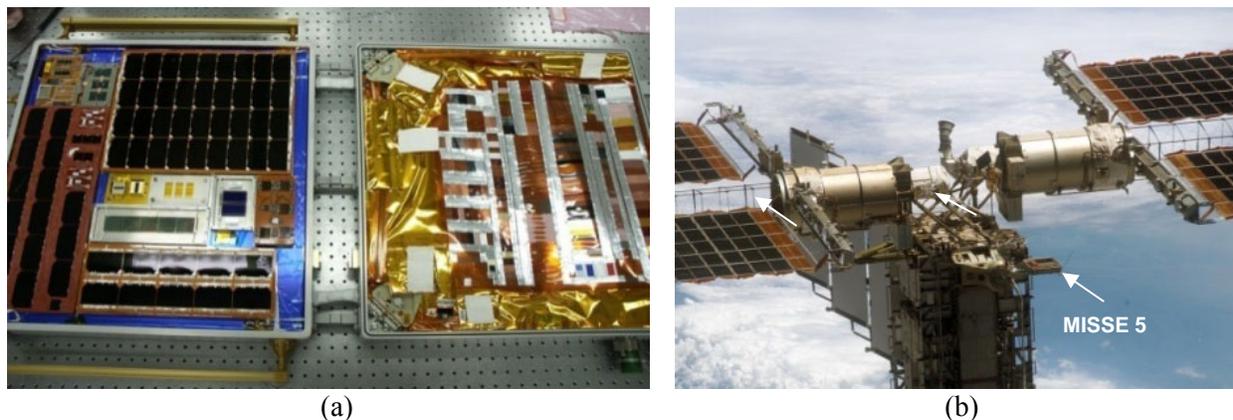


Figure 1.—MISSE 5: (a) Pre-flight, and (b) On-orbit photo taken during STS-114 of the zenith facing experiments.



Figure 2.—Pre-flight photograph of the MISSE 5 Thermal Blanket Experiment (before the stitching and additional tape was applied). The PEACE Polymer samples are outlined in white.

MISSE 5 PEACE Polymers Experiment

The MISSE 5 PEACE Polymers experiment consisted of 49 to 0.5 in. (1.27 cm) × 1.5 in. (3.81 cm) rectangular polymer material samples with 53 polymer materials. The majority of samples were thin film flexible polymers. A few rigid samples were also flown sandwiched between two pieces of either Kapton H or Kapton HN with Y966 acrylic adhesive. The primary experiment objective was to determine the atomic oxygen erosion yield (E_y , cm^3/atom) of polymers after space exposure in a nadir orientation. As the Thermal Blanket Materials Experiment needed to be thin and flexible, samples were taped and then sewn onto a Kapton blanket substrate therefore making determination of the E_y based on mass loss impossible. Therefore, all samples were dusted with fine salt-spray particles to provide isolated locations of protection from atomic oxygen erosion so that recession depth measurements could be made post-flight for E_y determination (Ref. 3). Figure 2 is a pre-flight photograph of the MISSE 5 Thermal Blanket Experiment with the PEACE Polymer experiment samples highlighted.

Thirty-seven of the 49 PEACE polymer samples were in a configuration that could be evaluated for surface embrittlement through bend-testing. The other 12 samples were not in thin film form and hence were not tested. A list of the flight samples that were tested along with the MISSE 5 flight sample number, the polymer name, the polymer abbreviation, trade names and the film thickness, are provided in Table 1 in the Results and Discussion section.

Experiment Procedures

Sectioning Bend-Test Samples

Because the MISSE 5 PEACE polymers were multi-purpose samples, only a portion of each sample was bend-tested for strain-induced surface cracking. A piece measuring 0.5 in. (1.27 cm) × 0.2 in. (0.508 cm) of each sample was sectioned for bend testing. The small salt particles were removed from the samples prior to bend-testing by carefully brushing off the dust with a small horse-hair water color brush. The salt was washed off a few samples (M-2, M-18, M-24, T-1, T-6) by rinsing with water and then gently drying the sample with pressurized nitrogen.

Bend-Test Procedures

The samples were analyzed for space-induced embrittlement using a bend-test procedure in which a surface strain was applied to the sample without adding overall tensile load. The strain necessary to induce surface cracking was determined by bending the samples over mandrels. Bend-testing was conducted using an apparatus with a semi-suspended pliable platform, and a set of mandrels varying in

diameter. A total of 23 mandrels were used, ranging in diameter from 1.253 to 0.052 cm. The diameter of each mandrel was calculated by using the mean diameter obtained by averaging four measurements made using Fowler & NSK Max-Cal electronic digital calipers. Each sample was bend-tested using successively smaller mandrels, with the sample being inspected after being bent around each mandrel. This procedure was continued until cracks were visible or until the sample did not experience any cracking with the smallest mandrel, in which case the sample was recorded as having not cracked.

During bend-testing, the sample was placed with its space-exposed face down onto the semi-guided apparatus. The mandrel was pushed down onto the sample, forcing the material to bend against a pliable surface that spanned two supports on the apparatus. The sample was bent in a U-shape, where the space-exposed surface was in tension and the backside surface was under compression. As the diameter of the mandrels decreased, the tension on the space-exposed surface of the sample increased because the sample was forced to bend more tightly around the mandrel.

Optical microscopy was used to document any surface features in the bend-test area prior to testing, and the same area was examined after bending around each mandrel to identify and document any induced surface cracks. The samples were examined at magnifications of approximately 10X to 13.8X with an Olympus SMZ stereo-zoom optical microscope outfitted with a Canon digital camera. An electronic coordinate system, the Boeckeler Microcode II Digital Readout, was used to find the same test location for microscopy examination before and after bend-testing at each mandrel, always centered at a dot that had been marked on the sample so that it would always be bent in the same location. Depending on the size of the sample, nine to fifteen optical microscopy pictures of each sample were taken before bending so that the entire bend-test sample was documented. When the sample was first observed to crack during the bend test process, the mandrel, and hence the strain, were recorded and the sample was not tested further. If no cracks could be detected under the optical microscope, the sample was bend-tested again with the next smaller mandrel, and the procedure was repeated. To verify the test results, several samples were bend-tested more than once.

Strain Calculations

Equation (1) allows the percent strain (E) to be calculated based on the thickness of the sample (t) and the diameter of the mandrel (d). This equation was derived from the bend-test configuration shown in Figure 3.

$$E = \left(\frac{t}{d+t} \right) \times 100 \quad (1)$$

The percent strain for each individual material, as stated before, was determined based on the mandrel at which it initially cracked and the thickness of the sample.

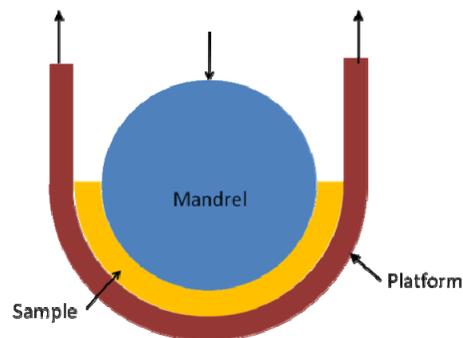


Figure 3.—Illustration of the bend-test configuration showing a cradle platform used to bend the sample around the mandrel.

Results and Discussion

Two of the PEACE Polymers samples were found to have cracked in half while on-orbit: Sample T-1 polymethyl methacrylate (PMMA, 50.8 μm thick) and Sample X-2 polymethylpentent (PMP, 50.8 μm thick). Close-up post-flight photos of Samples T-1 and X-2 are shown in Figures 4(a) and (b), respectively.

The set of photos in Figure 5 is an example of the microscope images obtained before and after bend-testing with each mandrel. The sample in Figure 5 is polyvinyl fluoride (PVF), also known as clear Tedlar (MISSE T-7). The box in each figure shows where cracks developed during bend-testing. This 25.4 μm thick sample cracked under a surface strain of only 0.38 percent.

While the PVF polymer shown in Figure 5 is representative of typical MISSE 5 PEACE polymer behavior, with the formulation of very small but visible vertical cracks, Figure 6 shows Sample X-2 (PMP), which exhibited more extreme behavior. This is one of two samples that cracked on-orbit. It was found to be extremely embrittled and fractured into pieces during bend-testing. In Figure 6 similar features are circled to facilitate the comparison process. Space exposure embrittled this polymer to a degree where the sample fell apart upon bend-testing with the largest mandrel (1.25 cm dia.), which provided a surface strain of only 0.4 percent. Sample T-1, PMMA, also cracked with the largest mandrel and fractured into pieces.

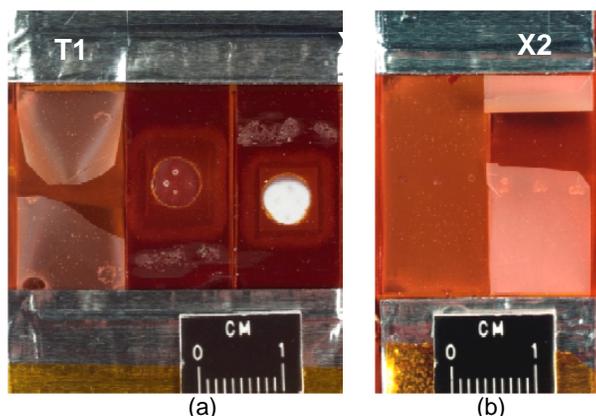


Figure 4.—Post-flight photographs of cracked PEACE samples: (a) Sample T-1, PMMA (left sample) and (b) Sample X-2, PMP (right sample).

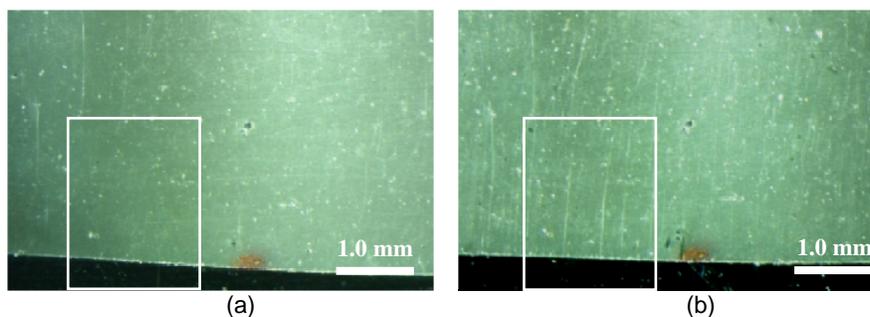


Figure 5.—Sample T-7, PVF: (a) before bend-testing and (b) after bend-testing.

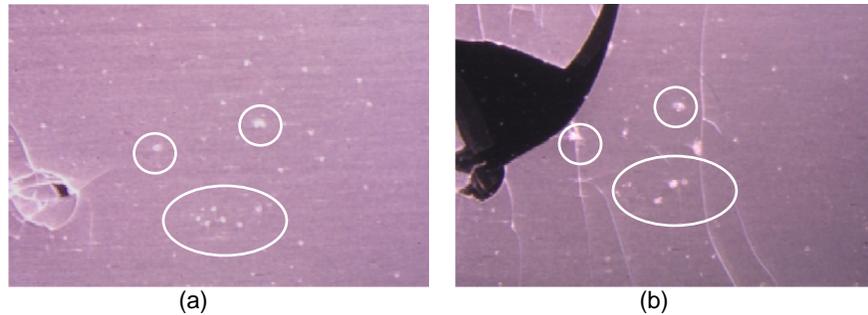


Figure 6.—Sample X-2, PMP: (a) before bend-testing and (b) after bend-testing.

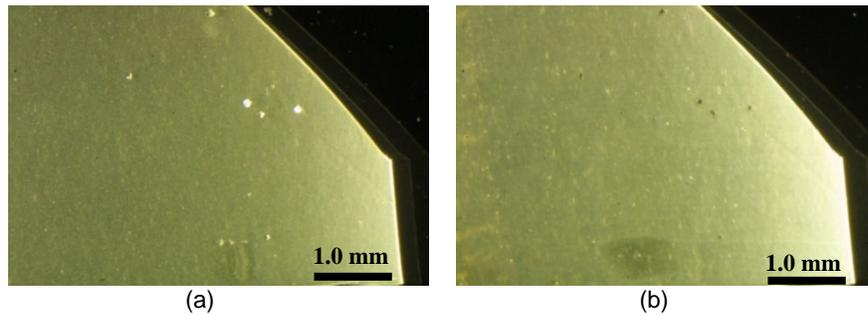


Figure 7.—Sample T-5, PU: (a) before bend-testing and (b) after bend-testing.

An example of a sample that did not crack during bend-testing, and hence remained ductile, is shown in Figure 7. This is Polyurethane (PU), MISSE 5 sample T-5. In summary, 17 of the 37 flight samples experienced some degree of surface cracking during bend-testing, while none of the pristine samples experienced any degree of surface cracking.

A list of the tested MISSE 5 PEACE samples, and their bend-test results, are provided in Table 1. In Table 1, DNC means Did Not Crack. It should also be noted that bright white materials such as Sample V-2, expanded polytetrafluoroethylene (ePTFE), would be particularly hard to see very fine surface cracks. Also, sample M-11, fluorinated ethylene propylene, appeared to contain surface scratches or cracks prior to bend testing, which may have impacted the bend-test results. Because the bend-test procedure relies on optical microscopy for identification of very small surface cracks, it is desirable to verify these bend-test results by examining the bend-tested samples with scanning electron microscopy or another technique that can verify the development of surface cracks.

The fact that surface strain induced cracking occurred in FEP (M-11), Kapton E (U-3), PTFE (V-3), and TOR (V-5), and that Upilex-S (M-14) did not crack, is consistent with results by Miller and Dever who characterized the tensile properties of these same polymers flown on the MISSE 5 Thermal Blanket Experiment as part of the Polymer Film Thermal Control Experiment (PFTC) (Ref. 4).

TABLE 1.—MISSE 5 BEND-TESTING RESULTS

MISSE ID	Material	Thickness, μm	Percent strain
M-02	Cellulose acetate (CA), Clarifoil	50.8	DNC
M-03	Polybutylene terephthalate (PBT), GE Valox 357	76.2	DNC
M-04	Chlorotrifluoroethylene (CTFE), Neoflon M-300	127	DNC
M-05	Crystalline polyvinylfluoride w/white pigment (PVF), White Tedlar TWH10BS3	50.8	DNC
M-07	Perfluoroalkoxy (PFA), Teflon PFA 500 LP	127	DNC
M-08	Tetrafluoroethylene-ethylene (ETFE), Tefzel ZM	76.2	4.26
^a M-11	Fluorinated ethylene propylene (FEP), Teflon FEP	50.8	2.09
M-13	Ethylene-chlorotrifluoroethylene (ECTFE), Halar 300	76.2	DNC
M-14	Polyimide BPDA (Upilex-S) (PI), Upilex-S 25S	25.4	DNC
M-16	Polyamide 6 (PA 6), Nylon 6	50.8	DNC
M-17	Polyamide 66 (PA 66), Nylon 66	50.8	5.61
M-18	Polyacrylonitrile (PAN), Barex 210	50.8	8.60
M-19	Polybenzimidazole (PBI), Celazole PBI 22	50.8	DNC
M-21	Poly(p-phenylene-2,6-benzobisoxazole) (PBO), Balanced Biaxial film	25.4	DNC
M-23	Polyetheretherketone (PEEK), Victrex PEEK 450	76.2	9.92
M-24	Polyethylene terephthalate (PET), Mylar A-200	50.8	0.76
M-25	Polyimide (CPI) (PI), CPI	76.2	1.51
Q-2	Tetrafluoroethylene-ethylene (ETFE), Tefzel 500 LZ	127	6.91
T-1	Polymethyl methacrylate (PMMA), Plexiglas	50.8	0.40
T-4	Polysulphone (PSU), Thermolux P1700	50.8	DNC
T-5	Polyurethane (PU), Duraflex PS S010	50.8	DNC
T-6	Polyvinylidene fluoride (PVDF), Kynar 740	76.2	2.16
T-7	Polyvinyl fluoride (PVF), Tedlar TTR10SG3 (clear)	25.4	0.38
U-1	Polyetherimide (PEI), Utem 1000	254	DNC
U-2	Amorphous Fluoropolymer (AF), Teflon AF 1601	50.8	1.45
U-3	Polyimide PMDA (PI), Kapton E	50.8	1.28
U-5	Ultra High Molecular Weight Polyethylene (UHMWPE)	254	DNC
U-7	Polyvinyl chloride (PVC), Clear-Lay Rigid PVC	127	DNC
V-1	Tetrafluoroethylene hexafluoro-propylene vinylidene fluoride (THV), Clariflex	254	25.72
^b V-2	Expanded polytetrafluoroethylene (ePTFE) (ISS cable material)	228.6	DNC
V-3	Polytetrafluoroethylene (PTFE), PTFE T-100 Virgin Skived Sintered Film	76.2	5.03
V-4	Polyimide (PI), Kapton 100 CB	127	DNC
V-5	Poly Arylene Benzimidazole (TOR)	38.1	0.46
V-6	Poly Arylene Benzimidazole (COR)	38.1	0.70
V-7	Polysulfone (PSO)	50.8	DNC
X-1	Polyethersulfone (PES)	76.2	DNC
X-2	Polymethylpentent (PMP)	50.8	0.40

^aFEP had cracks in the surface before bend-testing

^bePTFE is bright white and cracks may be hard to see

Summary and Conclusions

A study was conducted to characterize the embrittlement of thin film polymers after exposure to the space environment. In these investigations a bend test procedure was used to characterize the embrittlement of 37 different polymers that were exposed to LEO aboard the ISS for 13 months in a nadir-facing orientation as part of the MISSE 5 PEACE polymers experiment. Using mandrels of different diameters combined with optical microscopy, the strain necessary to induce surface cracking was determined for flight samples and compared with that of pristine samples. While none of the pristine materials cracked at the highest strain available, 18 of the 37 flight samples (49 percent) experienced embrittlement, shown by surface-tensile-induced cracking, after just 13 months of exposure in LEO. Two of the polymers, PMMA and PMP, had cracked while on-orbit and were so brittle that they fractured when bend-tested with the largest mandrel. These results indicate that many thin film polymers are susceptible to embrittlement in the LEO space environment, even after low solar and particle radiation exposures. Therefore, even “minimal” amounts of radiation exposure must not be overlooked when designing spacecraft components based on expected mechanical properties.

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