Materials International Space Station Experiment 5 Polymer Film Thermal Control Experiment

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It is known that polymer films can degrade as a result of space environmental exposure, but the magnitude of the mechanical property degradation and the degree to which the different environmental factors play a role is not well understood. An experiment was flown on the Materials International Space Station Experiment 5 to determine the change in tensile strength and percent elongation of some typical polymer films exposed in a nadir-facing environment on the International Space Station and, where possible, compare with similar ram- and wake-facing experiments flown on the Materials International Space Station Experiment 1 to get a better indication of the role the different environments play in mechanical property change. It was found that the majority of the polymers tested experienced some loss in tensile/yield strength and percent elongation with polytetrafluroethylene Teflon having the greatest change. Where comparisons could be made with the Materials International Space Station Experiment 1, it appears that the loss in percent elongation is dependent on the radiation level while the loss in tensile strength is not as sensitive to the level of radiation.

Introduction

THIN film polymers are used in many spacecraft applications for thermal control (multilayer insulation and sunshields), as lightweight structural members (solar array blankets, inflatable/deployable structures), and have been proposed for propulsion (solar sails). Polymers in these applications are exposed to the space environment and are vulnerable to degradation by solar ultraviolet radiation, solar flare X-rays, solar wind electrons and protons trapped in the Earth's magnetic field, temperature and orbital thermal cycling, micrometeoroids and orbital debris, and low-Earth-orbit atomic oxygen [1]. In applications where the polymer film is unsupported or is the structural member, it is important that the mechanical properties are not degraded beyond the limits set for its intended application.

The polymer film thermal control experiment (PFTC), first flown as one of many experiments on the Materials International Space Station Experiment (MISSE) 1, was designed to expose tensile specimens of a small selection of polymer films on ram-facing and nonram-facing surfaces of MISSE 1 [2]. A more complete description of the NASA John H. Glenn Research Center at Lewis Field MISSE 1-7 experiments is contained in a publication by de Groh et al. [3]. The PFTC was expanded and flown as one of the experiments on the nadir-facing side [side always pointing towards the Earth (gravitational direction)] of MISSE 5 in order to examine the long-term effects of the space environment on the mechanical properties of a wider variety of typical spacecraft polymers exposed to the nadir-facing space environment. A total of 33 tensile specimen samples (11 different types of three samples each) were flown on the MISSE 5 PFTC Experiment. The results of the post flight testing of these samples are described in this paper.

MISSE 5 Environment Description

MISSE 5 was placed on the aft P6 Trunion Pin Handrail of the International Space Station (ISS) by the crew of STS-114 on August

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3rd, 2005. The experiment was retrieved by the crew of STS-115 on 15 September 2006 after 13 months in space. Figure 1 shows a photo of the position of MISSE 5 on the ISS. Estimated environmental conditions provided by Finckenor and Pippin[‡] for the nadir side of MISSE 5 during deployment are given in Table 1. The estimated number of thermal cycles for MISSE 5 was about 6400. Temperature range was estimated from the experiment deck temperature for the Forward Technology Solar Cell Experiment on the solar-facing side of MISSE 5 [4].

PFTC Experiment Description, Apparatus, and Procedure

Description of Samples

The polymers that were exposed on MISSE 5 and evaluated for changes in mechanical properties are described in Table 2. Coated samples are indicated by a "/" separating each layer. The layers are listed in order from closest to farthest from the space-facing surface. The 8% PTFE-SiO_x (8% polytetrafluoroethylene–silicon oxide) coating is an ion beam cosputter deposited coating approximately 100 nm in thickness that was applied at NASA John H. Glenn Research Center at Lewis Field [5]. It was deposited from a silicon dioxide target with a PTFE wedge sized to give an 8% volume fraction of PTFE with the remainder silicon oxide. The coating was added to provide protection from the atomic oxygen environment in order to eliminate this environmental factor for better comparison. The coated Kapton E sample was received from the manufacturer with a vapor deposited aluminum (VDA) coating on the back of the sample of approximately 100 nm thickness for simulation of a backside surface reflective layer. It also had an Si coating of unknown thickness on the front side.

Tensile test specimens for flight and backup were fabricated from the polymer materials described in Table 2 using a die manufactured according to specimen "Type V" under the American Society for Testing and Materials (ASTM) Standard D-638 [6]. The dog-bone-shaped die had a gage length of 7.62 mm and an average gage width of $3.21\pm0.02\,$ mm. Three dog-bone-shaped tensile test samples of each polymer type were selected to be the flight samples and these were taped to a polyimide Kapton blanket that comprised the nadir viewing side of MISSE 5 along with other samples using aluminum tape at the edge of each grip end. The samples were then stitched to the blanket through the tape to firmly hold the samples in place for

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[†]Personal electronic mail communication with Miria Finckenor (NASA Marshall Space Flight Center) and telephone conversation with Gary Pippin (retired from Boeing), 13 Aug. 2009.

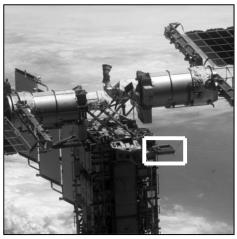


Fig. 1 Photograph of MISSE 5 location on the ISS taken by the STS-114 crew (MISSE 5 can be seen inside the boxed-in area).

flight. A photograph of the samples on the blanket is shown in Fig. 2. After retrieval, the tensile samples were carefully cut from the blanket near the tape line to remove them from the blanket but leave enough grip length for testing.

Tensile Testing

A DDL, Inc., Model 200Q bench-top tensile tester manufactured by TestResources, Inc., was used to test the MISSE 5 PFTC flight and control samples post retrieval. All of the samples were kept in the same controlled room environment with the tensile tester 48 h before testing to eliminate variation due to change in the environment as recommended by ASTM Standard Test Method for Tensile Properties of Thin Plastic Sheeting D882-02 and ASTM D-638 [6,7]. Tensile tests were conducted according to ASTM D-638 [6], using a 444.8 N load cell and a strain rate of 12.7 mm/ min. Each sample when loaded into the tensile holder was mounted in the grips with slack and then moved slightly with the motor drive to eliminate the slack without introducing initial tension on the sample. The initial grip separation was kept constant for all samples at 25.1 \pm 0.9 mm. Tests were conducted to obtain load-displacement data for each

Table 1 Summary of estimated environmental conditions for nadir side of MISSE 5

Environment	Dose
Atomic oxygen, atoms/cm ²	$\sim 1.8 \times 10^{20}$
Solar exposure (ESH)	165 ± 25 (direct)
•	360 ± 50 (Earth reflected)
	~525 (total)
Temperature, °C	~ -10 to $\sim +40$
•	\sim 6400 thermal cycles
Ionizing radiation, krads (Si)	\sim 2.75 dose through 127 μ m Kapton

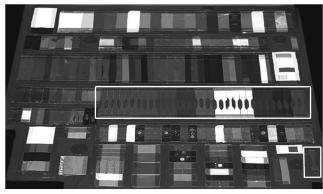


Fig. 2 Kapton blanket with samples exposed on nadir side of MISSE 5, PFTC experiment samples are outlined in white.

sample as well as the tensile (break at maximum load) or yield (yield at maximum load) strength [maximum load (N) divided by the original minimum cross-sectional area (m²) of the test sample] and the percent elongation at break (change in grip distance at break divided by the initial grip distance times 100).

Results and Discussion

Initial Observations

The PFTC flight samples were all intact with no evidence of tearing or breakage of the polymer post flight. There were, however, differences in some of the flight samples in comparison with the controls. The uncoated polymer flight samples were more matte in appearance indicating some atomic oxygen erosion on the surface and the 8% PTFE-SiO_x coated CP1 showed evidence of surface cracking of the coating. Examples of both post flight conditions are shown in Fig. 3. The remaining coated samples were very similar in appearance to their respective unflown control sample counterparts.

Load vs Displacement

Load vs displacement data was measured on three flight samples and three controls for each of the 11 materials tested unless otherwise noted. The majority of the samples exhibited a region of Hookean behavior where the load vs displacement was linear. Most of these materials also exhibited a clean break at peak load. Both the coated and uncoated PTFE Teflon also had a fairly linear load vs displacement but failed by developing a v-notch on one edge or the other within the gage length at a displacement near the point of failure which initiated a tear across the tensile specimen. Both the 8%PTFE-SiO_x coated CP1 and the TOR LM samples had nonlinear load vs displacement curves with a yield point at maximum load. The load at break was lower than the load at the yield point for these two materials. For the majority of the samples the slope of the load vs displacement curves for the control and flight samples were very close to each other near the break point. Figures 4 and 5 illustrate typical load vs displacement curves for both linear and nonlinear conditions, respectively.

Table 2 MISSE 5 PTFC Samples

Sample description and overall thickness	Polymer description	Polymer manufacturer
Teflon FEP (50.8 μm)	Fluorinated ethylene propylene	DuPont
8% PTFE-SiO _x /Teflon FEP (50.8 μ m)	Fluorinated ethylene propylene	DuPont
8% PTFE-SiO _x /Upilex S (25.4 μ m)	Aromatic polyimide	UBE Industries, Ltd.
8% PTFE-SiO _x /CP1 (25.4 μm)	Fluorinated polyimide	SRS Technologies
Kapton E (50.8 μ m)	Aromatic polyimide	DuPont
Si/Kapton E/VDA (50.8 μm)	Aromatic polyimide	DuPont
PTFE Teflon (76.2 μ m)	Polytetrafluoroethylene	Saint-Gobain
8% PTFE-SiO _x /PTFE Teflon (76.2 μ m)	Polytetrafluoroethylene	Saint-Gobain
Kapton HN (50.8 μ m)	Aromatic polyimide	DuPont
8% PTFE-SiO _x /Kapton HN (50.8 μ m)	Aromatic polyimide	DuPont
TOR LM (50.8 μm)	Polyarylene ether benzimidazole	Triton Systems, Inc.

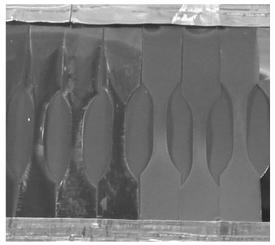


Fig. 3 8% PTFE-SiOx/CP1 (three samples on the left) showing evidence of cracking of the surface coating and uncoated Kapton E (three samples on the right) showing matte surface post flight.

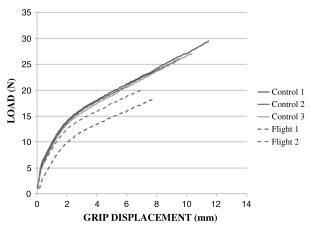


Fig. 4 Load vs displacement for uncoated polyimide Kapton HN comparing control with flight exposed samples. Curve exhibits mostly linear behavior near break point.

The flight exposed uncoated and coated PTFE Teflon samples were the only samples to show a significant change in the slope of the load vs displacement curves indicating a change in the elastic modulus of the PTFE Teflon with exposure. Figure 6 contains the load vs displacement curves for uncoated PTFE Teflon comparing

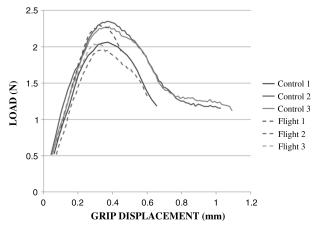


Fig. 5 Load vs displacement for TOR LM comparing control with flight exposed samples. Curve exhibits mostly nonlinear behavior near yield point.

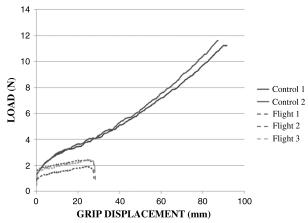


Fig. 6 Load vs displacement for uncoated PTFE Teflon comparing control with flight exposed samples illustrating a change in slope of the curve for the flight samples compared with the controls.

flight and control samples. The curves for the coated PTFE Teflon were nearly identical. All of the PTFE Teflon samples failed by tearing from one edge to the other across the width of the narrowest part of the test sample. The other polymer samples appeared to fail

Table 3 Tensile or yield strength comparison for samples flown on MISSE 5 and those kept on the ground as controls.^a

	Tensile or yield strength, mpa		
Sample description	Flight	Control	Percent loss
Teflon FEP (50.8 μm)	7.9 ± 1.2	14 ± 1	45 ± 10
8% PTFE-SiO _x /Teflon FEP (50.8 μ m)	10 ± 3	15 ± 2	30 ± 27
8% PTFE-SiO _x /Upilex S (25.4 μ m)	280 ± 100	220 ± 80	-31 ± 61
*8% PTFE-SiO _x /CP1 (25.4 μm)	7.7 ± 11	21 ± 4	64 ± 57
Kapton E (50.8 μ m)	120 ± 7	160 ± 8	22 ± 7
Si/Kapton E/VDA (50.8 μm)	150 ± 18	140 ± 12	-8.5 ± 15
Teflon PTFE (76.2 μ m)	9.3 ± 1.3	47 ± 2	80 ± 6
8% PTFE-SiO _x /Teflon PTFE (76.2 μm)	11 ± 0.5	41 ± 6	74 ± 17
Kapton HN (50.8 μ m)	120 ± 9	170 ± 14	41 ± 4
8% PTFE-SiO _x /Kapton HN (50.8 μ m)	140 ± 10	170 ± 10	18 ± 8
*TOR LM (50.8 μ m)	13 ± 1	14 ± 1	6 ± 12

^aDenotes samples with yield point before break.

Table 4 Percent elongation for samples flown on MISSE 5 and those kept on the ground as controls

	Pe	Percent elongation		
Sample description	Flight	Control	Percent loss	
Teflon FEP (50.8 μm)	150 ± 15	220 ± 7	33 ± 8	
8% PTFE-SiO _x /Teflon FEP	170 ± 31	230 ± 4	24 ± 14	
$(50.8 \ \mu m)$				
8% PTFE-SiO _x /Upilex S	13 ± 1	13 ± 2	-4 ± 15	
$(25.4 \ \mu m)$				
8% PTFE-SiO _x /CP1 (25.4 μm)	1.6 ± 2.2	3.3 ± 1.0	53 ± 75	
Kapton E (50.8 μ m)	22 ± 2	27 ± 0.9	20 ± 8	
Si/Kapton E/VDA (50.8 µm)	20 ± 3	18 ± 2	-13 ± 21	
Teflon PTFE (76.2 μ m)	110 ± 2	350 ± 12	68 ± 4	
8% PTFE-SiO _x /Teflon PTFE	110 ± 7	310 ± 28	63 ± 11	
$(76.2 \ \mu m)$				
Kapton HN (50.8 μ m)	29 ± 3	41 ± 4	29 ± 12	
8% PTFE-SiO _x /Kapton HN	34 ± 3	44 ± 3	21 ± 10	
$(50.8 \ \mu \text{m})$				
TOR LM (50.8 μm)	1.9 ± 0.5	3.7 ± 0.9	49 ± 30	

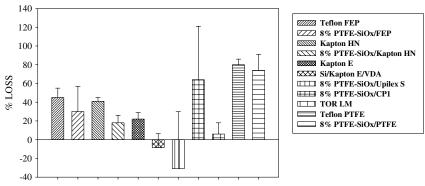


Fig. 7 Percent loss in tensile or yield strength for MISSE 5 polymers.

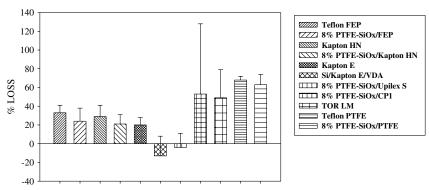


Fig. 8 Percent loss in elongation for MISSE 5 polymers.

more by breaking uniformly across the narrowest part of the test sample.

Tensile Strength and Percent Elongation

Control and flight comparison data (tensile or yield strength and percent elongation to break) for all sample types are contained in Tables 3 and 4 and the percentage loss of each property is graphically illustrated in Figs. 7 and 8. The data contained in the tables (flight, control and percent loss) have been rounded to two significant figures from the original tensile test data so the percent loss may not be in good agreement with the rounded table values but matches with the original data before rounding. As can be seen from the data, except for Si/Kapton E/VDA, coated Upilex S and TOR LM, all of the samples experienced some loss in tensile/yield strength. One of the uncoated Kapton flight samples experienced uneven loading during testing and was not used in the data average. Examination of the remaining data appears to exhibit a trend that coated polymers have lower loss in tensile/yield strength than their uncoated counterparts although the difference is within error of the measurement for both fluoronated ethylene propylene Teflon and PTFE Teflon. It is surmised that the higher loss in strength for some of the uncoated polymers may be due to stress centers developing from atomic oxygen texturing of the surface of the uncoated polymer. Similar trends were noticed for the loss in percent elongation of the polymers but in this case, except for Si/Kapton E/VDA all of the data were within error of each other.

Uncoated Kapton E experienced a loss in tensile strength about half the amount experienced by Kapton HN. The reduction in percent elongation for the uncoated Kapton E, however, was within error of that for uncoated Kapton HN. The Si/Kapton E/VDA samples did not show a statistically significant change in tensile strength or elongation indicating that the coating protected the Kapton E from being affected by the environment. There was a great deal of variation in the coated Upilex S samples. The error was much larger than the change in tensile strength or the change in percent elongation for these samples so it is difficult to draw any meaningful conclusions. The samples had a mottled appearance as if the polymer were a mixture rather than a uniform polymer film.

The coated CP1 samples and the TOR LM samples both broke at lower stress than their yield points. These samples were again mottled in appearance and had wide variation in the data. Both the CP1 and TOR LM samples had very low percent elongation to break. Two of the CP1 flight samples broke while in the grips just before the load was applied. The TOR LM percent elongation was reduced upon flight exposure but the yield strength change was within the error of the measurement. The opposite is true for the coated CP1 sample. That data indicated a reduction in yield strength but the error in the measurement for the percent elongation was larger than the change.

Coated and uncoated PTFE Teflon experienced the greatest loss in tensile strength (\sim 74% and \sim 80% respectively) and elongation (\sim 63% and \sim 68% respectively). The presence of a coating did not appear to play a significant role in the change indicating that atomic

Table 5 Comparison of environmental conditions on MISSE 1 and MISSE 5

Environment	MISSE 5 (nadir)	MISSE 1 (ram)	MISSE 1 (wake)
Atomic oxygen, atoms/cm ² Solar exposure (ESH)	~1.8 × 10 ²⁰ ~525	$\sim 8.5 \times 10^{21}$ $5400-6400$	$\sim 2.0 \times 10^{20}$ $4500-5600$
Temperature, °C	\sim - 10 to +40 6400 thermal cycles \sim 2.75 dose through	\sim - 30 to +40 22,800 thermal cycles \sim 11 dose through	\sim - 30 to +40 22,800 thermal cycles \sim 11 dose through
Ionizing radiation, krads (Si)	\sim 2.73 dose inrough 127 μ m Kapton	\sim 11 dose through 127 μ m Kapton	\sim 11 dose through 127 μ m Kapton

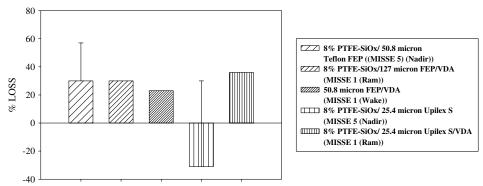


Fig. 9 Comparison of percent loss in tensile strength between polymers flown on MISSE 1 and MISSE 5.

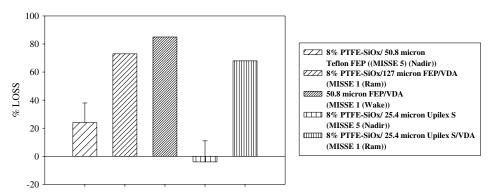


Fig. 10 Comparison of loss in percent elongation between polymers flown on MISSE 1 and MISSE 5.

oxygen did not play a large role in the loss in mechanical properties for this polymer.

Comparison with MISSE 1 Data

As shown in Table 5, MISSE 1 experienced nearly $4 \times$ more thermal cycles over roughly the same temperature range and nearly a $4\times$ higher radiation dose than MISSE 5 [2]. There was also \sim 11× more equivalent sun hours (ESH) of vacuum ultraviolet radiation (VUV) on the ram side of MISSE 1 and \sim 9.6× more ESH of VUV radiation on the wake side of MISSE 1 than on the MISSE 5 nadir viewing side [2]. Unfortunately not too many of the tensile samples flown on MISSE 1 could be compared with those flown on MISSE 5 because of sample breakage on MISSE 1 and some differences in the types of samples flown. CP1 was flown on both experiments but the polymer was from different lots and the initial (control) yield strength and percent elongation were very different (about $4 \times$ and $2 \times$ higher for the MISSE 1 control than the MISSE 5 control, respectively) so it was difficult to make direct comparisons. The only polymers that could be compared were coated Upilex S and coated and uncoated FEP Teflon. Figures 9 and 10 graphically illustrate a comparison of the MISSE 1 and MISSE 5 data for these polymers for both percent loss in tensile strength and percent loss in elongation.

Upilex S flown on the ram side of MISSE 1 experienced a 36% loss in tensile strength and a 68% loss in elongation [2]. The spread in the MISSE 5 tensile strength data is very large so it could be similar to the MISSE 1 results. The control mechanical properties are within error of each other. The percent elongation data for MISSE 5, however, had less variation and experienced a negligible change in comparison with the 68% loss on MISSE 1. The data for the loss in mechanical properties for MISSE 1 and MISSE 5 for coated FEP Teflon were again within error of each other. The percentage loss in tensile strength for the MISSE 1 sample was 30% [2] in comparison with ~30% on MISSE 5. So essentially there was no difference in loss of tensile strength between the two very different environments. It may be that the environment affects the tensile strength for FEP Teflon up to a limit and beyond the limit, there are no further changes. There was, however, a difference in percent elongation between the

MISSE 5 and MISSE 1 samples. The MISSE 1 coated FEP sample had a 73% loss in elongation [2] compared with the $\sim\!\!24\%$ loss on MISSE 5 which is about a factor of 3 difference. It is interesting to note that the uncoated FEP Teflon flown on the wake side of MISSE 1 had very similar values for loss in tensile strength and percent elongation (23 and 85%, respectively) as on MISSE 1 ram side [2] in spite of the difference in VUV illumination. MISSE 5 had a much lower VUV radiation dose, but it also had a lower number of thermal cycles and less ionizing radiation than MISSE 1. It appears that either there is a VUV damage limit or other environmental factors than VUV radiation dose play the largest role in the loss in mechanical properties for FEP Teflon. Further testing separating out each environmental factor is needed in order to determine which constituent of the environment or combination is causing the greatest damage.

Conclusions

The majority of the samples flown on MISSE 5 experienced some loss in tensile or yield strength and percent elongation as a result of exposure to the environment except for Si/Kapton E/VDA and 8% PTFE-SiO_x/Upilex S which had larger variation in the sample measurements than the percent loss that was calculated. Protected Kapton HN, Kapton E and FEP Teflon all had similar losses in percent elongation to their uncoated counterparts. All of these coated samples, however, had less of a loss in tensile strength than their uncoated counterparts, although for FEP Teflon it was within the error of the measurement. Since the Kapton samples had noticeable surface texture due to atomic oxygen erosion but no significant thickness loss, it is possible that the texture may give rise to stress points on the surface that cause the samples to break at a lower peak load. The greatest loss in tensile strength and elongation was exhibited by the uncoated and coated PTFE Teflon samples. In this case, failure was dominated by some other component of the environment than atomic oxygen since both the coated and uncoated PTFE experienced nearly the same losses. Comparing MISSE 1 and MISSE 5 test results indicated that the loss in tensile strength for the coated FEP Teflon samples was independent of the VUV and

radiation levels or number of thermal cycles indicating that there may be a damage limit which MISSE 1 and 5 both exceeded for this property. The percent loss in elongation, however, was greater for coated Upilex S and coated FEP Teflon flown on MISSE 1 showing that there is an environmental exposure dependence for this property. The levels at which changes occur, and which environment factor or combination of factors causes these changes is unclear and needs further investigation in experiments where these factors can be controlled or eliminated independently.

Acknowledgments

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