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Thermal Contributions to the Degradation of Ground Laboratory and Space-Irradiated Teflon®

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The aluminized Teflon® fluorinated ethylene propylene outermost layer of the multilayer insulation blankets on the Hubble Space Telescope has become embrittled, resulting in severe on-orbit cracking. During the second servicing mission, a piece of aluminized fluorinated ethylene propylene was retrieved. This cracked piece of insulation had curled on-orbit, exposing the backsurface aluminum to space. This extremely embrittled piece reached 200°C on orbit, 150°C higher than the nominal temperature extreme. Therefore, experiments have been conducted to determine the effect of heating on the degradation of fluorinated ethylene propylene that has been irradiated in a ground laboratory facility or in space on the Hubble Space Telescope. Samples of pristine, x-ray irradiated and Hubble Space Telescope retrieved fluorinated ethylene propylene were heat treated from 50 to 200°C at 25°C intervals in a high vacuum facility and tensile tested. Density measurements were also obtained. Results indicate that heating does not embrittle nonirradiated Teflon. However, there is a significant dependence of the embrittlement of irradiated Teflon on heating temperature, with near complete loss of elongation at 100°C and higher. Rate of degradation changes, which were consistent with the glass transition temperature for fluorinated ethylene propylene, were present in the data. The results support chain scission as the primary mechanism of degradation of fluorinated ethylene propylene on the Hubble Space Telescope and indicate the significance of the on-orbit temperature of fluorinated ethylene propylene with respect to its degradation in the space environment.

Nomenclature

T_g = glass transition temperature, °C
α_s = solar absorptance
ε = thermal emittance
ρ = density, g/cm^3

Introduction

THE Hubble Space Telescope (HST) was launched on 25 April 1990 into low Earth orbit as the first mission of NASA's Great Observatories program. The HST is a telescope capable of performing observations in the near-ultraviolet, visible, and near-infrared wavelengths (0.115–2.5 μm). The HST was designed to be serviced on orbit to upgrade scientific capabilities. Four servicing missions have taken place to date. A fifth mission is planned for 2005.

The Hubble Space Telescope is covered with two primary types of thermal control materials, radiators and multilayer insulation (MLI) blankets, which passively control temperatures on orbit.[1] Both of these thermal control materials utilize metallized Teflon® fluorinated ethylene propylene (FEP) as the exterior (space-facing) layer. Metallized Teflon FEP is a common thermal control material used on spacecraft, but it has been found to degrade in the low-Earth-orbital (LEO) space environment. Teflon FEP is used as the outer layer of thermal control insulation because of its excellent optical properties (low solar absorptance α_s and high thermal emittance ε), in addition to its flexibility. A metallized layer (Al or Ag) is applied to the backside of the FEP to reflect incident solar energy. The α_s and ε of 5-mil (127-μm)-thick FEP with an aluminized backing are 0.13 and 0.81, respectively.[2] Solar radiation (UV radiation and x-rays from solar flares), electron and proton radiation (omnidirectional particles trapped in the Van Allen belts), thermal exposure and thermal cycling, and atomic-oxygen exposure are all possible LEO environmental factors that could possibly contribute to the degradation of FEP.

Analyses of aluminized-FEP (Al-FEP) and silvered-FEP (Ag-FEP) MLI blankets retrieved from HST during the first servicing mission (SM1) after 3.6 years of space exposure revealed that the 5-mil (127-μm)-thick FEP exterior layer was embrittled on high solar exposure surfaces.[3,4] Surfaces that received the highest solar exposures had microscopic through-thickness cracks in the FEP at stress locations.[3,4] Bonded solar facing 2-mil (51-μm) Al-FEP on the solar-array drive arm (SADA) power harness, which was also retrieved during SM1, had many cracks and experienced a total loss of mechanical integrity in heavily stressed areas.[5] The maximum temperature during thermal cycling of the power harness FEP was higher (>130°C) (Ref. 5) than that of the MLI FEP (maximum temperature of 50°C) (Ref. 6).

During the second servicing mission (SM2), after 6.8 years in space, severe cracking of the 5-mil Al-FEP MLI outer layer was observed on the light shield (LS), forward shell, and equipment bays of the telescope. Astronaut observations combined with photographic documentation revealed extensive cracking of the MLI in many locations, with solar facing surfaces being heavily damaged.[2] Figure 1a shows two large cracked areas on the LS. A very large vertical crack can be seen, and a smaller cracked area, in which free-standing Al-FEP had curled up tightly (with the FEP surface in compression), is located above the vertical crack. A close up of the tightly curled Al-FEP is shown in Fig. 1b. The worst of the MLI outer layer cracks were patched during SM2. Prior to patching the upper LS crack, the tightly curled Al-FEP outer layer was cut off and retrieved for postmission analyses. Patches of 5-mil (127-μm)-thick Al-FEP were placed over the two LS cracks, and patches of 2-mil (51-μm)-thick Al-FEP were placed over cracks in MLI on equipment bays 8 and 10. Figure 2 shows one of these cracked areas from bay 10. As determined through an HST MLI Failure Review

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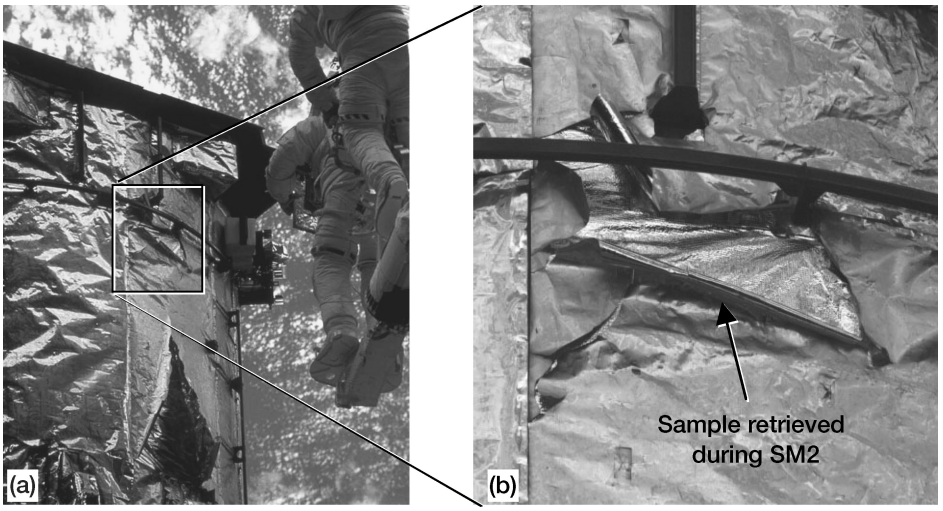

Fig. 1 MLI damage on the HST as witnessed during SM2: a) two cracked areas in the LS MLI outer layer, a large vertical crack and above it a tightly curled area, and b) close-up of the tightly curled Al-FEP prior to retrieval.

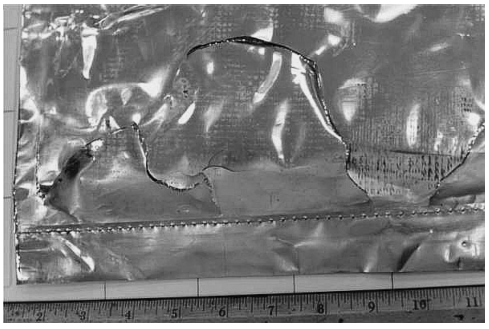

Fig. 2 Through-thickness cracks in 5-mil Al-FEP outer layer MLI from bay 10, photographed after retrieval during SM3A.

Board, embrittlement of FEP on HST is caused by radiation exposure (electron and proton radiation with contributions from solar flare x rays and UV radiation) combined with thermal cycling.[6] It is not known whether atomic oxygen in the Hubble environment enhanced the radiation-induced embrittlement process.

During SM3A, original MLI from Bay 10, which experienced 9.7 years of space exposure, as well as 2-mil-thick Al-FEP patch material, which experienced 2.8 years of exposure, were retrieved and available for degradation analyses. Postretrieval analyses have shown that FEP retrieved during SM2 after 6.8 years of exposure was more embrittled, with 0% elongation to failure, than FEP retrieved 2.8 years later during SM3A, after 9.7 years of exposure.[7,8] Because the retrieved SM2 material curled with the FEP surface in compression exposing the lower emittance Al surface to space, it experienced a higher temperature extreme during thermal cycling ($\approx$200°C) than the nominal solar-facing MLI experiences ($\approx$50°C). As this was the only sample retrieved during SM2, it was important to determine the difference between the measured properties of this excessively heated sample and those of nominally heated MLI FEP. The 2-mil-thick Al-FEP thermal shields that covered the solar-array bistems of the second pair of HST solar arrays (SA-2) thermal cycled to a maximum temperature of 130°C on orbit. Because of the various temperatures experienced by HST FEP materials, it is necessary to understand the effect of temperature on FEP degradation on HST. Understanding temperature effects is important for determining degradation mechanisms and for facilitating the prediction of FEP degradation in LEO.

Prior investigations have been conducted by de Groh et al.[7,9] on the effects of heating (at 130 and 200°C) pristine FEP and FEP irradiated in ground facilities or in LEO. It was desired to get additional data at temperatures between room temperature and 200°C. Therefore, samples of pristine FEP, x-ray irradiated FEP, and SM3A-retrieved FEP were heated at temperatures from 50 to 200°C at 25°C intervals in a high vacuum furnace and evaluated for changes in tensile properties and density in order to improve the understanding of the degradation of this insulation material in the LEO space environment. X rays were used for the source of irradiation because x rays from solar flares are believed to contribute to the embrittlement of FEP on HST[6] and because previous ground tests have shown that solar flare x rays are energetic enough to cause bulk embrittlement in 127-μm FEP,[10] simulating the type of damage that occurs on HST. Also, the mechanism of embrittlement of Teflon is believed to be similar for electron and x-ray radiation, and x rays do not cause charge related problems. Therefore, x-ray exposure is a useful technique for understanding radiation damage effects in Teflon.

Materials

Pristine FEP and FEP for X-Ray Irradiation

Teflon FEP is a perfluorinated copolymer of tetrafluoroethylene and hexafluoropropylene (HFP). The FEP used for x-ray irradiation followed by vacuum heat treatment and for nonirradiated heat treatment tests was nonaluminized 5 mil thick (127 μm).

HST SM3A Al-FEP

The HST FEP used in this study was retrieved by astronauts from equipment bay 10 during servicing mission SM3A. Figure 3a shows a close-up of the MLI and patches on bay 10 prior to being removed from HST. Four different materials were retrieved: original MLI from the top section of bay 10 [top MLI (TM)], original MLI from the bottom section of bay 10 [bottom MLI (BM)], and patches installed during SM2 over portions of TM and BM, designated as top patch (TP) and bottom patch (BP), respectively. The outermost layer of the TM and BM was 5-mil-thick Al-FEP. The retrieved patch material was 2-mil-thick Al-FEP. The Al was $\approx$1000 Å for these materials.

Samples were sectioned from various regions of the BM, TP, and BP surfaces for postflight analyses, and the results were reported elsewhere.[7,8,11] For the BM surface, regions designated as R1 and R2 refer to the areas without a patch and covered by a patch, respectively. A large section of the original MLI material, which was not covered by a patch and thus had been exposed to the space environment for 9.7 years (section BM-R1), was cut from the MLI blanket and provided for these heating studies. This sample section can be seen in Fig. 3b. Because this large sample was sectioned from the blanket in 2001, whereas most samples for postflight analyses testing were sectioned shortly after the December 1999 flight in 2000, this particular sample is referred to as the SM3A 2001 BM-R1 sample. The 2001 BM-R1 sample was carefully examined, and

Table 1 Exposure conditions for retrieved HST FEP materials

Exposure	SM2 FEP[12] (LS, +V3)	SM3A BM-R1 MLI[7] (bay 10, −V2)
On-orbit duration, years	6.8	9.7
Thermal cycles/temperature range, cycles/°C	37,100/−100–+50 −100–+200 when curled	52,550/−100–+50
Equivalent solar hours (ESH)	<33,638	13,598
X-ray fluence, J/m^2	1–8 Å[a]: <209 0.5–4 Å[a]: <13	1–8 Å: 62 0.5–4 Å: 3.9
Electron fluence, #/cm^2, >40 keV	$<1.95 \times 10^{13}$	2.74×10^{13}
Proton fluence, #/cm^2, >40 keV	$<1.95 \times 10^{10}$	2.77×10^{10}
Atomic-oxygen fluence, atoms/cm^2	$<3.2 \times 10^{20}$	$<5.7 \times 10^{20}$

[a]Values reported in Ref. 12 incorrectly assumed that +V3 surfaces always face direct sun.

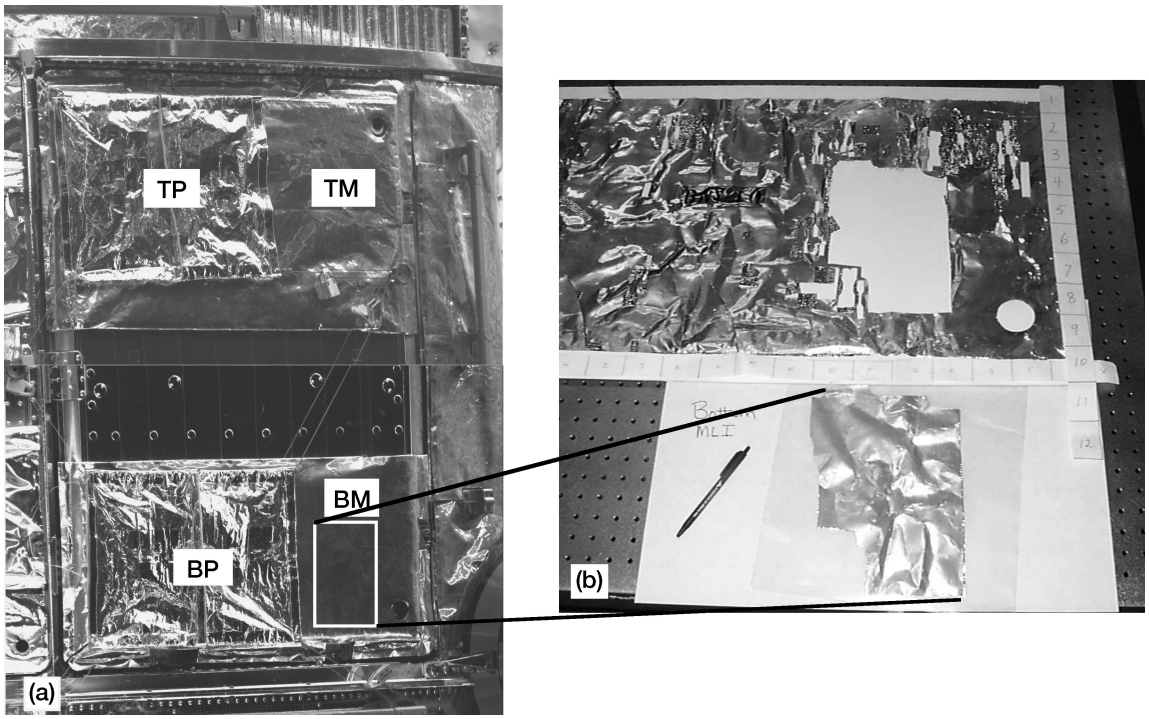

Fig. 3 HST equipment bay 10 materials: a) bay 10 MLI and patches photographed during SM3A prior to removal from HST and b) a section of MLI blanket showing the location and approximate size of the SM3A 2001 BM-R1 sample.

the location of all impact sites and cracks were documented in order to avoid these areas when punching out tensile samples or cutting density samples.

HST Environment

Table 1 provides the space environmental exposure conditions for the retrieved SM2 and SM3A BM-R1 materials. For the samples, the table lists the direction the surface faces with respect to the coordinate system for HST, indicated by V2 and V3 axes (described next). The sample retrieved during SM2 faced the +V3 direction, which is the solar-facing surface of the Hubble. Because this section had cracked and curled with the FEP surface on the inside of the curl at some point during the mission, all environmental exposure conditions (except thermal cycling) are indicated as being some amount less than the values calculated for the entire mission duration. Bay 10 faces the −V2 direction (solar-array drive arm direction). The environmental exposure conditions of solar exposure hours, solar event x-ray fluence, electron and proton fluence, and atomic-oxygen fluence for FEP surfaces on HST vary depending on the direction the surface was facing and the position of nearby obstructing surfaces, whereas the number of thermal cycles is independent of direction.

Because the bay 10 MLI surfaces, which approximately faced the −V2 direction, were at an oblique angle to the sun, bay 10 MLI retrieved at SM3A actually received less equivalent solar exposure than the SM2 sample retrieved 2.8 years earlier. However, the SM3A BM-R1 MLI experienced many more thermal cycles, higher electron and proton radiation, and higher atomic-oxygen fluence than the SM2 sample. The exposure levels for various retrieved materials are affected by the solar activity, and there was a solar minimum in the time period between the SM2 and SM3A. More details of environmental exposures are provided in Refs. 8 and 12.

Experimental Procedures

X-Ray Exposure (Pristine FEP)

A modified high-vacuum x-ray photoelectron spectroscopy facility was used to irradiate the pristine FEP tensile samples. A copper target was irradiated with a 15.3-kV, 30-mA electron beam producing Cu x rays (Cu Kα at 8048 eV, Cu L at 930 eV). The tensile samples were located 30.5 mm from the target, and the Cu x rays were filtered through a 2-μm Al window (part of the x-ray tube). Samples were irradiated at a high vacuum pressure of 10^{-8} torr. A 25-mil (635-μm)-thick beryllium filter was placed over the FEP samples to absorb the low energy Cu L components, which would contribute significantly to damaging only the surface.[13] The x-ray flux was 13.28 W/m^2 (Ref. 14). The target material, electron beam energy, and filter were chosen to produce a high-flux, uniform distribution of energy absorbed vs depth in the film. The energy deposition

rate, or dose rate, vs depth below the surface for 127-μm FEP film at the specified exposure conditions are provided by de Groh and Gummow.[15] The technique used to characterize the x-ray source and energy deposition within the FEP film is described by Pepper and Wheeler.[13] Pepper et al.[14] provide quantitative characterization of the Cu x-ray source and the absorbed energy deposition rate within a 75-μm film. X-ray irradiated samples were stored under vacuum until they were tensile tested or vacuum heat treated.

The x-ray exposure was not intended to simulate the full extent of damage occurring on the Hubble, but to cause irradiation-induced polymer damage with still enough elongation at failure remaining to see the effects caused by heating. Based on a series of prior tests, it was determined that a 2-h exposure would provide the desired reduction in tensile properties.[15] Prior tests also indicated that the maximum number of samples which could be uniformly exposed at a time was two. The samples were centered in a holder that provided a 2.0×2.0 cm exposure area. (The tensile sample gauge length is $\approx$1 cm.) Density tests were conducted on pieces sectioned from a 200°C heated irradiated tensile sample and indicated that the x-ray exposure was uniform across the length of the exposed area. The total energy absorbed per unit area integrated through the full thickness (the areal dose D) of the 127-μm film for the 2-h exposure was 33.8 kJ/m^2 (3.38 J/cm^2) (S.V. Pepper, NASA Glenn Research Center, personal communication, 1999).

Vacuum Heat Treatment

Pristine FEP, x-ray-exposed FEP, and HST SM3A Al-FEP samples were vacuum heat treated from 50 to 200°C at 25°C intervals in a high vacuum facility adapted with a tube furnace. A Teflon-lined Cu pipe was placed inside the tube furnace to promote uniform heating. The exposure temperature was monitored with a thermocouple attached to a Teflon witness sample, held in contact with the test samples. The pressure was 10^{-6} to 10^{-7} torr during heating. Samples were heated for a target (minimum) time of 72 h.

Tensile Properties

Samples for tensile testing were "dog-bone" shaped and die cut using a tensile specimen die manufactured according to ASTM D638-95, type V. The tensile samples were 3.18 mm wide in the narrow section (neck), with a 9.5-mm gauge length. Samples were tested using a bench-top tensile tester with a 4.54-kg load cell and a test speed of 1.26 cm/min. Ultimate tensile strength (UTS) and elongation at failure were determined from the load displacement data.

Density Measurements

Density measurements were obtained using density gradient columns calibrated using glass float standards of known densities (± 0.0001 g/cm^3). The density solvents used were carbon tetrachloride (CCl_4, $\rho = 1.594$ g/cm^3) and bromoform ($CHBr_3$, $\rho = 2.899$ g/cm^3). The presence or absence of the thin (1000-Å) aluminized coating (as removed by NaOH solution) was found to have no effect on the density of the Al-FEP samples.[9]

Results and Discussion

Room-Temperature Tensile Properties

Pristine FEP

The room-temperature (23°C) tensile data for pristine FEP are listed in Table 2. The UTS and percent elongation at failure for 13 pristine FEP samples were 24.1 ± 1.5 MPa and 271.2 ± 16.9%, respectively.

HST SM3A Al-FEP

The tensile results for the as-retrieved SM3A FEP are listed in Table 2. As can be seen, the retrieved SM3A Teflon from HST, after 9.7 years in the space environment, is substantially degraded. If compared to the pristine FEP tested in this study, the UTS of the retrieved HST FEP has decreased from 24.1 to 13.9 MPa, and the elongation at failure has decreased from 271.2 to 55.3% because of the space environment exposure. These correspond to decreases of 42.3 and 79.6%, respectively. These tensile properties provide insight into the damage mechanism of Teflon in space. Because the UTS decreased with the elongation at failure of the space-exposed FEP, chain scission is identified as the dominant degradation mechanism of the FEP on HST.

X-Ray-Irradiated FEP

After x-ray exposure, the UTS and percent elongation at failure for 10 samples were 17.1 ± 1.5 MPa and 212.7 ± 31%, respectively, as seen in Table 2. The x-ray-irradiation-induced embrittlement corresponds to a 29.0% reduction in the UTS and a 21.6% reduction in percent elongation at failure compared to pristine FEP.

Table 2 Tensile properties of vacuum heat-treated pristine, x-ray-irradiated and HST-retrieved Teflon FEP

Vacuum heat treatment temperature	Material	Number of samples	UTS, MPa	Elongation at failure, %
Room temperature 23°C	Pristine FEP	13	24.1 ± 1.5	271.2 ± 16.9
	X-ray FEP	10	17.1 ± 1.5	212.7 ± 31
	HST Al-FEP	4	13.9 ± 0.4	55.3 ± 9.3
50°C (±1°C)	Pristine FEP	4	23.4 ± 0.7	264.1 ± 12.6
	X-ray FEP	4	15.1 ± 1.1	162.6 ± 35.5
	HST Al-FEP	4	13.9 ± 0.3	46.5 ± 4.1
75°C (±2°C)	Pristine FEP	6	22.5 ± 1.5	259.6 ± 14.2
	X-ray FEP	8	15.3 ± 0.2	134.9 ± 46.3
	HST Al-FEP	4	14.4 ± 0.1	25.7 ± 5.5
100°C (±1°C)	Pristine FEP	4	22.1 ± 0.7	250.2 ± 5.7
	X-ray FEP	4	15.5 ± 0.3	43.1 ± 6.6
	HST Al-FEP	4	14.5 ± 0.2	10.4 ± 1.1
125°C (±1°C)	Pristine FEP	4	22.5 ± 0.8	254.7 ± 8.3
	X-ray FEP	4	15.8 ± 0.2	23.8 ± 4.9
	HST Al-FEP	3	14.2 ± 0.7	9.4 ± 3.3
150°C (±3°C)	Pristine FEP	4	22.8 ± 0.6	271.0 ± 6.4
	X-ray FEP	4	15.4 ± 0.3	22.8 ± 5.6
	HST Al-FEP	4	14.2 ± 0.3	8.7 ± 4.2
175°C (±12°C)	Pristine FEP	3	22.1 ± 0.8	287.4 ± 11.1
	X-ray FEP	4	16.4 ± 0.8	15.2 ± 3.5
	HST Al-FEP	4	14.9 ± 0.8	7.9 ± 2.0
200°C (±4°C)	Pristine FEP	4	23.7 ± 0.8	306.8 ± 9.6
	X-ray FEP	2	16.1 ± 0.4	9.7 ± 5.2
	HST Al-FEP	3	14.5 ± 0.5	4.5 ± 0.9

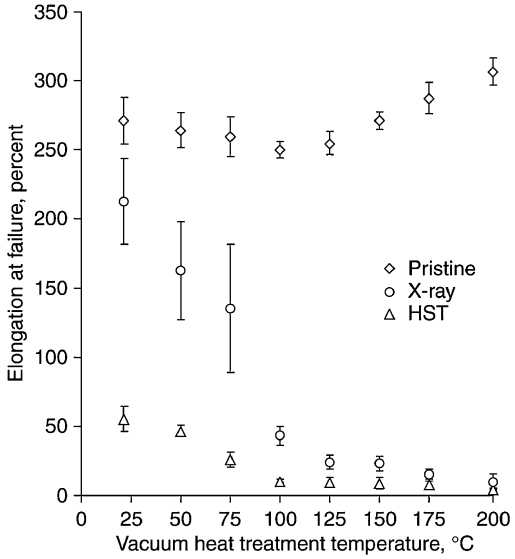

Fig. 4 Percent elongation at failure of pristine, ground laboratory x-ray-exposed, and retrieved HST FEP as a function of vacuum heat treatment temperature.

Vacuum Heat Treatment

Tensile Properties

The results of tensile tests for the pristine, ground-laboratory-irradiated, and HST FEP after vacuum heat treatment are listed in Table 2 along with the room-temperature data. The elongation at failure data are graphed in Fig. 4. There was no degradation in the tensile properties of vacuum heat-treated, nonirradiated FEP. In fact, this batch of FEP showed an increase in the percent elongation at failure after higher temperature exposures (175 and 200°C). Although vacuum heat treatment did not cause much additional change in the UTS of x-ray-irradiated FEP, there was a dramatic decrease in the percent elongation at failure, as can be seen in Fig. 4. The elongation decreased from 212.7% at 23°C to only 9.7% after 200°C exposure, corresponding to a 95% decrease in elongation. As can be seen in the graph, there is a rapid decrease in the elongation with heating from 23 to 100°C, with near complete losses of elongation at 125°C and above. Only two of the original four x-ray samples heated to 200°C could be tensile tested because two stuck together during heating and then broke during separation.

Although the FEP retrieved from HST was significantly embrittled in its as-retrieved condition, it became even more embrittled with vacuum heat treatment (even at temperatures already experienced on orbit). The space-exposed HST FEP followed a trend similar to that of the ground-laboratory x-ray-irradiated FEP, showing little changes in UTS and decreases in elongation from 23 to 100°C, with near complete loss of elongation with heating at 100°C and higher.

Density

The density data for the pristine, ground-laboratory-irradiated and HST space-irradiated FEP, at room temperature and after vacuum heat treatment, are listed in Table 3. The data are graphed in Fig. 5. The standard deviation is given when more than one sample was measured. As can be seen in the graph, the density of the retrieved HST FEP is essentially the same as pristine FEP, and the room-temperature x-ray-irradiated FEP is just slightly more dense than pristine FEP, even though these irradiated samples are significantly embrittled. These results indicate that although irradiation induces scission in the polymer chains, resulting in embrittlement, the actual packing of the chains is not affected by irradiation exposure.

There were very gradual increases in the density with heating up to 75°C for all samples. Significant increases started at 100°C, with larger increases corresponding to higher temperatures. Although the density increased with temperature for all samples, larger increases occurred for the samples that had been irradiated than for

Table 3 Density data of vacuum heat-treated pristine, x-ray-irradiated and HST-retrieved Teflon FEP

	Pristine FEP		X-ray FEP		HST FEP (SM3A 2001 BM-R1)	
Temperature, °C	Density, g/cm^3	Std. dev.	Density, g/cm^3	Std. dev.	Density, g/cm^3	Std. dev.
23	2.1373	0.0011	2.1407	——	2.1376	0.0005
50	2.1379	——	2.1414	——	2.1376	0.0005
75	2.1379	——	2.1428	——	2.1389	0.0005
100	2.1393	——	2.1477	——	2.1407	——
125	2.1414	——	2.1585	——	2.1456	——
150	2.1473	——	2.174	——	2.1577	——
175	2.1507	——	2.1775	——	2.1647	0.0016
200	2.1631	——	2.1856	——	2.1696	0.0031

Table 4 XRD and thermal data for as-retrieved and heated 5-mil HST FEP[7]

	XRD	Thermal characterization	
Sample	Crystallinity, %	Onset of melt, °C	Heat of fusion, mcal mg^{-1}
Pristine Al-FEP	28.2 ± 0.6	244.6 ± 3.0	-4.09 ± 0.47
Al-FEP 200°C heated	36	246	−4.96
SM2 (200°C on orbit)	46–47	236.2 ± 5.7	-6.70 ± 0.1
SM3A R1	28	243.7 ± 1.2	-4.20 ± 0.41
SM3A R1 200°C heated	45	218	−6.34

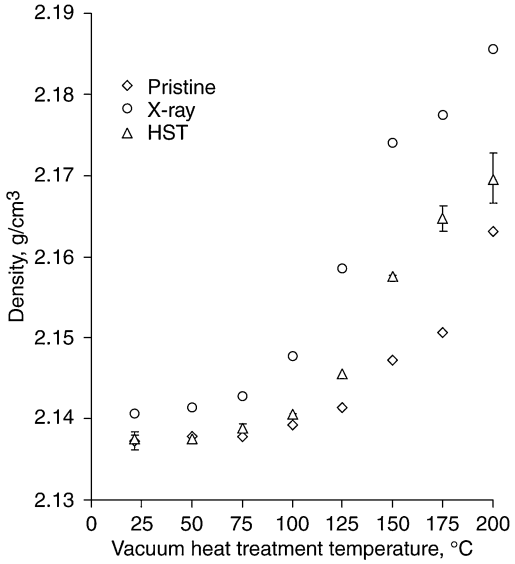

Fig. 5 Density of pristine, ground laboratory x-ray-exposed, and retrieved HST FEP as a function of vacuum heat treatment temperature.

pristine FEP. These results are consistent with de Groh's previous studies[7,9] that show the density of pristine FEP increases with heating, but FEP from HST has greater increases in density for the same heat treatment (200°C exposure). Greater density increases in the heated Hubble FEP is attributed to irradiation-induced scission of bonds in space, which allows for greater mobility and crystallization upon heating than that which occurs with nonirradiated FEP. X-ray diffraction (XRD) studies verify that increases in FEP density correlate to increases in polymer crystallinity.[7,9] Table 4 provides XRD crystallinity data for as-retrieved and 200°C heated HST FEP along with pristine FEP.[7] The density results further support chain scission as the primary mechanism of degradation of FEP in the space environment.

When comparing the curves for the elongation and density data, it was observed that in each set of data there appeared to be a noticeable change in the slope of the data around 100°C. The data were therefore graphed with linear fits for two sections of the data. The

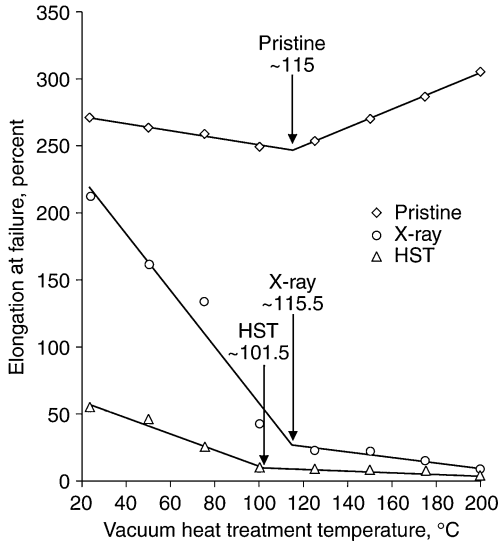

Fig. 6 Change in the slope of the percent elongation at failure data of pristine, ground laboratory x-ray-exposed, and retrieved HST FEP as function of vacuum heat treatment temperature.

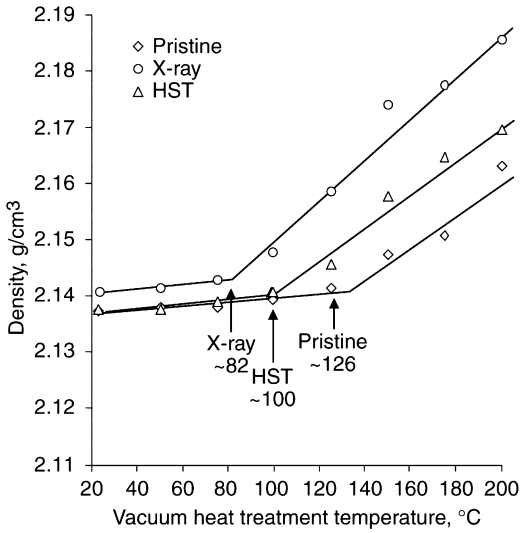

Fig. 7 Change in the slope of the density data of pristine, ground laboratory x-ray-exposed, and retrieved HST FEP as a function of vacuum heat treatment temperature.

lines chosen were based on the best fit for each individual section of data. The resulting curves for the elongation at failure and density data are shown in Figs. 6 and 7, respectively. The change-of-slope temperature has been highlighted in these graphs at the intersection of the two linear fits. The change-of-slope temperature of the pristine FEP (115°C for the elongation data and 126°C for the density data) correlates well with the glass transition temperature T_g (α relaxation), which is listed from ≈83–150°C in the literature, depending on HFP content.[9] Eby and Wilson report transition temperatures for FEP with densities (2.135–2.136 g/cm^3) similar to the pristine FEP examined in this report to be ≈150 and ≈127°C for 10.7 and 17.7 mol% HFP, respectively.[16] Commercially available FEP is reported to be 20 mol% HFP (Don Farrelly, DuPont, personal communication, 1999), which would indicate that the transition temperature for pristine FEP would be close to 125°C based on the Eby study, which is consistent with the change-of-slope temperatures for the pristine FEP. Another interesting observation is that the irradiated samples have lower change-of-slope temperatures, and hence lower T_g than pristine FEP. For example, the temperature in which the density of the ground-laboratory-irradiated FEP starts to increase quickly is 82°C, while it is 100°C for the HST retrieved FEP and 126°C for pristine FEP. These results indicate that irradiation causes changes in the polymer structure allowing thermal-induced increases in crystallization to occur at a lower temperature than which it occurs in pristine FEP. This observed shift of T_g toward lower values further indicates that the degradation of FEP is controlled by chain scission reactions. For linear polymers, there is a linear relation between the glass and melting temperatures. Because the melting temperature of the irradiated FEP was found to decrease (as will be shown in the following section) along with the T_g, FEP is found to preserve its linear character during degradation.

The irradiation-induced reduction in transition temperature appears to be consistent with previous thermal characterization studies, which indicate that there is a decrease in the onset of melt temperature (also know as the melting temperature) in FEP after irradiation exposure and heating. Differential scanning calorimetry thermal data for HST SM2 and SM3A samples and pristine FEP are provided in Table 4 along with the XRD data. The onset of melt and heat of fusion for the as-retrieved SM3A R1 FEP was very similar to that of pristine Al-FEP. The SM2 FEP, which had been heated to 200°C on orbit, had a much lower onset of melt and higher heat of fusion than pristine FEP. Heating pristine FEP caused no change in the onset of melt, but did cause an increase in the heat of fusion, indicating that bond scission did not occur with heating (indicated by a lowering of the onset of melt) but that the polymer had become more crystalline (indicated by an increase in the heat of fusion). This is consistent with the heated density and crystallinity data. Very significant decreases in the onset of melt and increases in the heat of fusion were observed for the SM3A R1 sample that was heated in air at 200°C, as shown in Table 4. These data imply that the SM3A R1 sample experienced more chain scission (lower onset of melt), but less increased crystallinity (smaller increase in the heat of fusion) than the SM2 sample. Perhaps the higher crystallinity of the SM2 sample (consistent with the XRD data) occurred because it was heated at 200°C on orbit during irradiation exposure and was heated for much longer than the SM3A sample. (The SM2 sample cycled to 200°C possibly for years.)

The DSC data showed that although the heated pristine FEP had an increase in heat of fusion the FEP exposed to the HST radiation environment underwent significant changes in both the heat of fusion and the onset of melt temperature with on-orbit heating or with ground laboratory heating. The results are consistent with the density and crystallinity data indicating similarities in properties of the embrittled as-retrieved nominally heated HST FEP (SM3A) as compared with pristine FEP, with significant differences observed after heating the space-exposed FEP.

Ground Laboratory and Space Irradiation Comparison

Although it was not the goal of this study to try to simulate the extent of damage on HST with the x-ray exposure, it was decided to compare the areal dose for x-ray-irradiated FEP with that experienced by the FEP on HST. The areal dose for the various irradiation exposures of 5-mil-thick HST SM3A FEP is provided in Table 5 (Joyce Dever, NASA Glenn Research Center, personal communication, 2002). The total areal dose for the SM3A BM-R1 FEP was 427.2 J/m^2. The 2001 SM3A BM-R1 FEP has an elongation at failure of 55% at the time it was tested with an areal dose of only 427 J/m^2, whereas the x-ray-exposed FEP was much less embrittled (213% elongation) after orders of magnitude higher areal dose (33,800 J/m^2). HST-exposed FEP received many additional environmental exposure conditions beyond x-ray irradiation, as shown in Table 1. The factors that could contribute the differences between space-exposed and ground-laboratory-irradiated FEP include the

Table 5 Areal dose for HST SM3A FEP

SM3A BM-R1 MLI (bay 10, −V2)	Areal dose, J/m^2
X rays, 1–8 Å	29.80
X rays, 0.5–4 Å	0.72
Electrons, >40 keV	389.6
Protons, >40 keV	7.11

extreme differences in the dose rates (i.e., time factor), the variation in ionizing species and energies, temperature differences during irradiation exposure, contributions from solar ultraviolet radiation and thermal cycling, and, possibly, surface effects from atomic oxygen.

Knowing from this research the importance of temperature on degradation, it would be better to compare the elongation of the HST FEP with the x-ray-exposed FEP heated to 50°C, the maximum on-orbit service temperature of the nominal HST FEP. The x-ray-exposed FEP heated to 50°C has an elongation of 162%, which is 60% relative to pristine FEP. Although this is somewhat closer to the degradation on HST, it is still much less embrittlement with orders of magnitude more areal dose. This stresses the difficultly in conducting simulated space environment durability tests and emphasizes the potential complication when conducting durability testing based strictly on expected mission fluence or dose values.

Conclusions

The objective of this research was to determine the effects of heating on ground-laboratory-irradiated fluorinated ethylene propylene (FEP) and FEP retrieved from the Hubble Space Telescope in order to better understand the effect of temperature on the rate of degradation, and on the mechanism of degradation, of this insulation material in the low-Earth-orbit environment. Samples of pristine FEP, x-ray-irradiated FEP, and HST SM3A-retrieved FEP were heated from 50 to 200°C at 25°C intervals in a high vacuum furnace and evaluated for changes in tensile properties and density. Results indicate that although heating does not degrade the tensile properties of nonirradiated Teflon there is a significant dependence on the degradation of the percent elongation at failure of irradiated Teflon as a function of heating temperature, with dramatic degradation occurring at 100°C and higher exposures. The density of nonheated irradiated FEP (ground or space irradiated) was essentially the same as pristine FEP, although these samples are significantly embrittled. This indicates that irradiation induces scission in the polymer chains, resulting in embrittlement, but chain packing is not affected. Gradual increases in the density occurred with heating from 23 to 75°C for all samples, with significant increases occurring at 100°C and higher exposures. Larger increases occurred for the irradiated samples than for the pristine FEP. These results were consistent with previous studies that show pristine FEP increases in density with heating, but irradiated FEP experiences greater increases for the same heat treatment. This is attributed to irradiation-induced scission of bonds, which allows for greater mobility and crystallization upon heating than that which occurs with nonirradiated FEP. Changes in the rate of degradation were present in both elongation and density data. The change-of-slope temperatures of pristine FEP (115°C for elongation and 126°C for density) correlate with the glass transition temperature of FEP. The change-of-slope temperature of irradiated FEP was lower than for pristine FEP, further indicating that scission damage has occurred. The tensile results and heated density data support chain scission as the dominant mechanism of degradation of FEP in the space environment. The results show the significance of the on-orbit service temperature of FEP with respect to its degradation.

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