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The Effect of Heating on the Degradation of Ground Laboratory and Space Irradiated Teflon® FEP

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July 2002

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THE EFFECT OF HEATING ON THE DEGRADATION OF GROUND LABORATORY AND SPACE IRRADIATED TEFLON® FEP

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Abstract

The outer most layer of the multilayer insulation (MLI) blankets on the Hubble Space Telescope (HST) is back surface aluminized Teflon® FEP (fluorinated ethylene propylene). As seen by data collected after each of the three servicing missions and as observed during the second servicing mission (SM2), the FEP has become embrittled in the space environment, leading to degradation of the mechanical properties and severe on-orbit cracking of the FEP. During SM2, a sample of aluminized-FEP was retrieved from HST that had cracked and curled, exposing its aluminum backside to space. Because of the difference in optical properties between FEP and aluminum, this insulation piece reached 200°C on-orbit, which is significantly higher than the nominal MLI temperature extreme of 50°C. This piece was more brittle than other retrieved material from the first and third servicing missions (SM1 and SM3A, respectively). Due to this observation and the fact that Teflon thermal shields on the solar array bi-stems were heated on-orbit to 130°C, experiments have been conducted to determine the effect of heating on the degradation of FEP that has been irradiated in a ground laboratory facility or in space on HST. Teflon FEP samples were x-ray irradiated in a high vacuum facility in order to simulate the damage caused by radiation in the space environment. Samples of pristine FEP, x-ray irradiated FEP and FEP retrieved from the HST during SM3A were heat treated from 50 to 200°C at 25° intervals in a high vacuum facility and then tensile tested. In addition, samples were tested in a density gradient column to determine the effect of the radiation and heating on the density of FEP. Results indicate that although heating does not degrade the tensile properties of non-irradiated Teflon, there is a significant dependence of the percent elongation at failure of irradiated Teflon as a function of heating temperature. Irradiated Teflon was found to undergo increasing degradation in the elongation at failure as temperature was increased from room temperature to 200°C. Rate of degradation changes, which were consistent with the glass I transition temperatures for FEP, appeared to be present in both tensile and density data. The results indicate the significance of the on-orbit temperature of Teflon FEP with respect to its degradation in the low Earth orbital space environment.

1.0 Introduction

The HST was launched on April 25, 1990 into low Earth orbit as the first mission of NASA's Great Observatories program. It is a telescope capable of performing observations in the near-ultraviolet, visible and near-infrared wavelengths (0.115 to 2.5 μm). The HST was designed to be serviced on-orbit to upgrade scientific capabilities. SM1 occurred in December 1993, after 3.6 years in space. SM2 was in February 1997, after 6.8 years in space. SM3A was in December 1999, after 9.7 years in space. The fourth servicing mission, designated as SM3B, occurred in March 2002. A future servicing mission is currently planned for mid 2003.

The HST is covered with two primary types of thermal control materials, radiators and multilayer insulation blankets, which passively control temperatures on-orbit.[1] Both of these thermal control materials utilize metallized Teflon FEP as the exterior (space-facing) layer. Metallized Teflon FEP is a common thermal control material used on spacecraft, such as the Long Duration Exposure Facility, the Solar Max Mission spacecraft and HST, 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 and low molecular weight. 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 (ultraviolet (UV) radiation and x-rays from solar flares), electron and proton radiation (omni-directional particles trapped in the Van Allen belts), thermal exposure and thermal cycling, and atomic oxygen exposure are all possible LEO environmental factors which could possibly contribute to the degradation of FEP.

Analyses of aluminized-FEP (Al-FEP) and silvered-FEP (Ag-FEP) MLI blankets retrieved during SM1 revealed that the 5 mil (127 μm) thick FEP exterior layer was embrittled on high solar exposure surfaces.[3,4] Surfaces which 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 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)[5] than that of the MLI FEP (maximum temperature of 50°C)[6].

During SM2, 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 1 shows two large cracked areas on the LS. A very large vertical crack can be seen near the center of the photograph, 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. 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 post-mission analyses. Patches of 5 mil thick (127 μm) Al-FEP were placed over the two LS cracks, and patches of 2 mil thick (51 μm) 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 a HST MLI Failure Review 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]

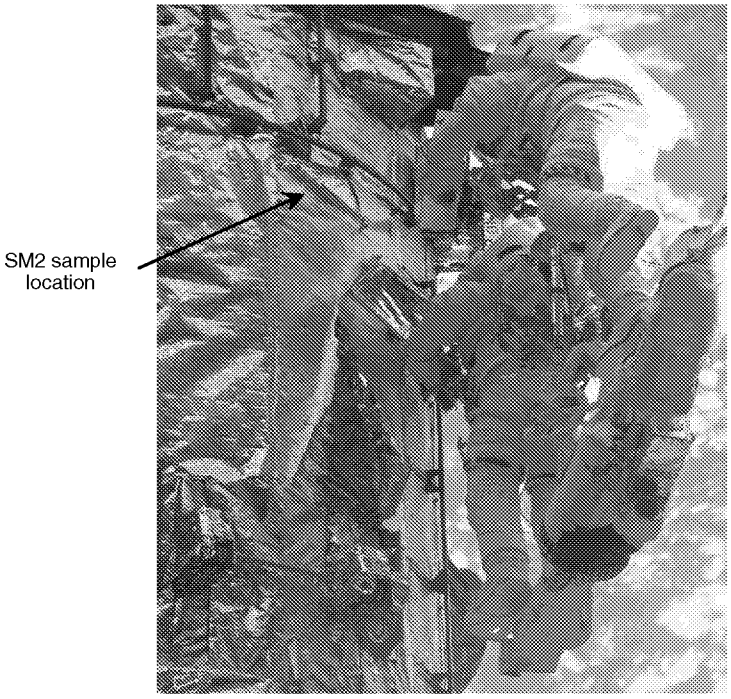

Figure 1. Two cracked areas in the MLI outer layer on the HST LS as witnessed during SM2. The astronauts have cut off the curled upper LS material and are preparing to place a patch over the area.

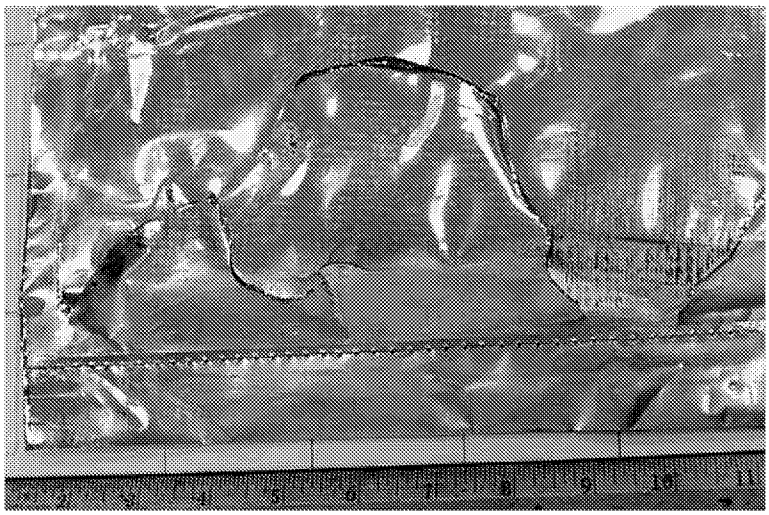

Figure 2. Cracks present in 5 mil thick Al-FEP Bay 10 MLI, photographed after retrieval during SM3A.

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. Post-retrieval analyses have shown that FEP retrieved during SM2 after 6.8 years exposure was more embrittled, with a 0% elongation to failure, than FEP retrieved 2.8 years later during SM3A, after 9.7 years 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 (∪200 ϒC) than the nominal solar facing MLI experiences (∪50 ϒC). 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 nominally heated MLI FEP. Another back surface metallized Teflon insulation, 2 mil thick Al-FEP thermal shields that covered the solar array bi-stems 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.

Investigations have been conducted by de Groh et al. on the effects of heating pristine FEP and FEP irradiated in ground facilities or in LEO, as reported in references 7 and 9. For this study, samples of pristine FEP, x-ray irradiated FEP and SM3A-retrieved FEP were heated at temperatures of 50°C to 200°C in 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-ray energies are energetic enough to cause bulk embrittlement in 127 μm FEP.[10] Also, the mechanism of embrittlement of polymers is believed to be the same for all forms of ionizing radiation, therefore x-ray exposure is a very useful technique for understanding radiation damage effects in Teflon.

2.0 Materials

2.1. PRISTINE FEP AND FEP FOR X-RAY IRRADIATION

Teflon® FEP is a perfluorinated copolymer of tetrafluoroethylene (TFE) and hexafluoropropylene (HFP). The FEP material used for x-ray irradiation followed by vacuum heat treatment and for non-irradiated heat treatment tests was non-aluminized 5-mil thick (127 μm), and was purchased from Sheldahl (lot #96-16).

2.2. HST SM3A Al-FEP

As previously mentioned, MLI blankets originally installed on HST on Bay 10 (exposed to the space environment for 9.7 years) and 2 mil Al-FEP patches installed on Bay 10 during SM2 (exposed for 2.8 years) were retrieved by astronauts 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 outer most layer of the TM and BM materials was 5 mil thick Al-FEP. Retrieved patch material was 2 mil thick Al-FEP. The Al was ≈1000 Å.

Samples were sectioned from various regions of the BM, TP, and BP surfaces for post-flight analyses and the results are reported in references 7, 8 and 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 Figure 3b. Because this large sample was sectioned from the blanket in 2001, while most samples for post-flight 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 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.

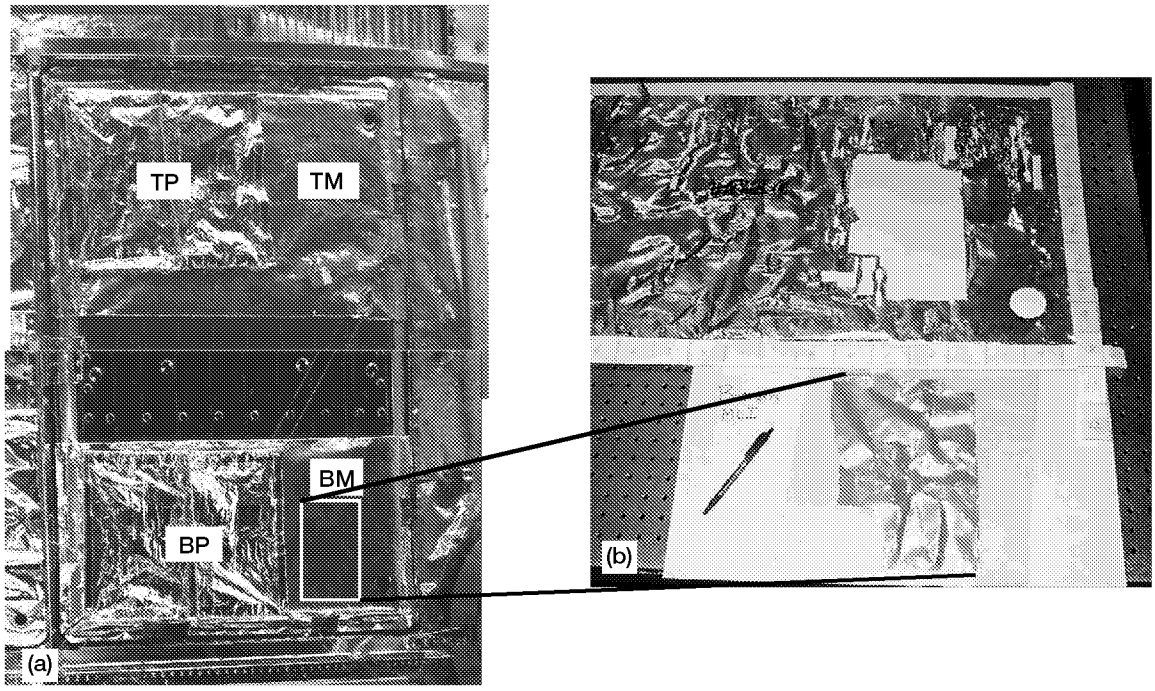

Figure 3. HST Equipment Bay 10 material: (a) Bay 10 MLI and patches during SM3A prior to removal from HST, and (b) A section of MLI blanket showing the location and size of the SM3A 2001 BM-R1 sample.

3.0 The 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 below). The sample retrieved during SM2 faced the +V3 direction, which is the solar facing surface of Hubble. Because this area 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 (SADA 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 position of nearby obstructing surfaces, whereas the number of thermal cycles is independent of direction.

Table 1. Exposure Conditions for Retrieved HST FEP Materials.

Exposure	SM2 FEP (LS, +V3)[12]	SM3A BM-R1 MLI[7] (Bay 10, –V2)
Thermal Cycles/Temperature Range (cycles/°C)	37,100/–100 to +50°C –100 to +200°C when curled	52,550/ –100 to +50°C
Equivalent Solar Hours (ESH)	< 33,638	13,598
X-ray Fluence (J/m^2)	1-8 Å*: < 209 0.5-4 Å*: < 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}

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

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, and higher electron and proton radiation, and atomic oxygen fluence than the SM2 sample. The exposure levels for various retrieved materials are affected by the solar activity, and it should be noted that there was a solar minimum between the SM2 to SM3A time period. More details of environmental exposures are provided in references 8 and 12.

4.0 Experimental Procedures

4.1 X-RAY EXPOSURE (PRISTINE FEP)

A modified X-ray photoelectron spectroscopy (XPS) 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). 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.[14] The choice of target material, electron beam energy, and filter was chosen to produce a high flux, uniform distribution of energy absorbed, versus depth in the film. The energy deposition rate, or dose rate, versus depth below the surface for 127 μm FEP film at the specified exposure conditions are provided by de Groh and Gummow in reference 15. The technique used to characterize the x-ray source and energy deposition within the FEP film is described by Pepper and Wheeler in reference 13. Pepper et al. provide quantitative characterization of the Cu x-ray source and the absorbed energy deposition rate within a 75 μm film in reference 14. 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 Hubble, but to cause irradiation induced polymer damage, and still have enough elongation at failure remaining to see the effects due to heating. Based on a series of prior tests, it was determined that a 2-hour exposure would provide the desired reduction in tensile properties.[15] Prior tests also indicated that the maximum number of samples that could be uniformly exposed at a time was two. The samples were centered in a holder that provided a 2.0 x 2.0 cm exposure area (the tensile sample gauge length is ≈ 1 cm). The total energy absorbed per unit area integrated through the full thickness (the areal dose, D) of the 127 μm film for the 2-hour exposure was 33.8 kJ/m^2.[16]

4.2 VACUUM HEAT TREATMENT

Pristine FEP, x-ray exposed FEP and HST SM3A Al-FEP samples were vacuum heat treated from 50°C to 200°C in 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 time of 72 hours.

4.3 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.

4.4 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, ρ = 1.594 g/cm^3) and bromoform ($CHBr_3$, ρ = 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.

5.0 Results and Discussion

5.1 ROOM TEMPERATURE TENSILE PROPERTIES

5.1.1 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, was 24.1 ± 1.5 MPa and 271.2 ± 16.9%, respectively.

5.1.2 HST SM3A Al-FEP

The retrieved SM3A Teflon from HST, after 9.7 years in the space environment, is substantially degraded. The tensile results for the as-retrieved SM3A FEP are listed in Table 2. 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%. 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 decrease in elongation at failure of the space-exposed FEP, chain scission is identified as the primary degradation mechanism on HST.

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

5.1.3 X-Ray Irradiated FEP

After x-ray exposure, the UTS and percent elongation at failure for 10 samples was 17.1 ± 1.5 MPa and 212.7 ± 31%, respectively. This is a 29.0% reduction in the UTS and a 21.6% reduction in percent elongation at failure due to irradiation embrittlement. 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.

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 5 mil thick HST SM3A FEP is provided in Table 3.[17] The total areal dose for the SM3A BM-R1 FEP was 427.2 J/m^2. It should be noted that the HST FEP has an elongation at failure of 55% with an areal dose of only 427 J/m^2, while the x-ray exposed FEP was much less embrittled (213% elongation), after orders of magnitude higher areal dose (33,800 J/m^2). The factors that could contribute to these differences include the *extreme* differences in the dose rates (i.e., time factor), the variation in ionizing species and energies, temperature differences during irradiation exposure and the contribution from thermal cycling on HST (52,550 cycles from –100 to +50°C), and possibly, surface effects from atomic oxygen and UV exposure in space. 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.

Table 3. Areal Dose for HST SM3A FEP.

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

5.2 VACUUM HEAT TREATMENT

5.2.1 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 data are graphed in Figure 4. There was no degradation in the tensile properties of vacuum heat-treated non-irradiated FEP, in fact, with this batch of FEP an increase in the percent elongation at failure was observed for the higher temperatures (175 and 200°C). Although heat treatment did not cause much change in the UTS of x-ray irradiated FEP with vacuum heat treatment, there was a dramatic decrease in the percent elongation at failure, as can be seen in Figure 4. The elongation decreased from 212.7% at 23°C to only 9.7% after 200°C exposure. This corresponds to a 95% decrease. And as can be seen in the graph, there is a rapid decrease in the elongation from 23°C to 100°C, with near complete losses of elongation from 125°C to 200°C. Only two of the original four x-ray samples heated to 200°C could be tensile tested because two stuck together slightly 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 after vacuum heat treatment at 50°C, the maximum on-orbit temperature). The space-exposed HST FEP followed a similar trend as the ground-laboratory x-ray irradiated FEP, showing little changes in UTS and decreases in elongation from 23°C to 100°C, with near complete loss of elongation with heating at 100°C and higher.

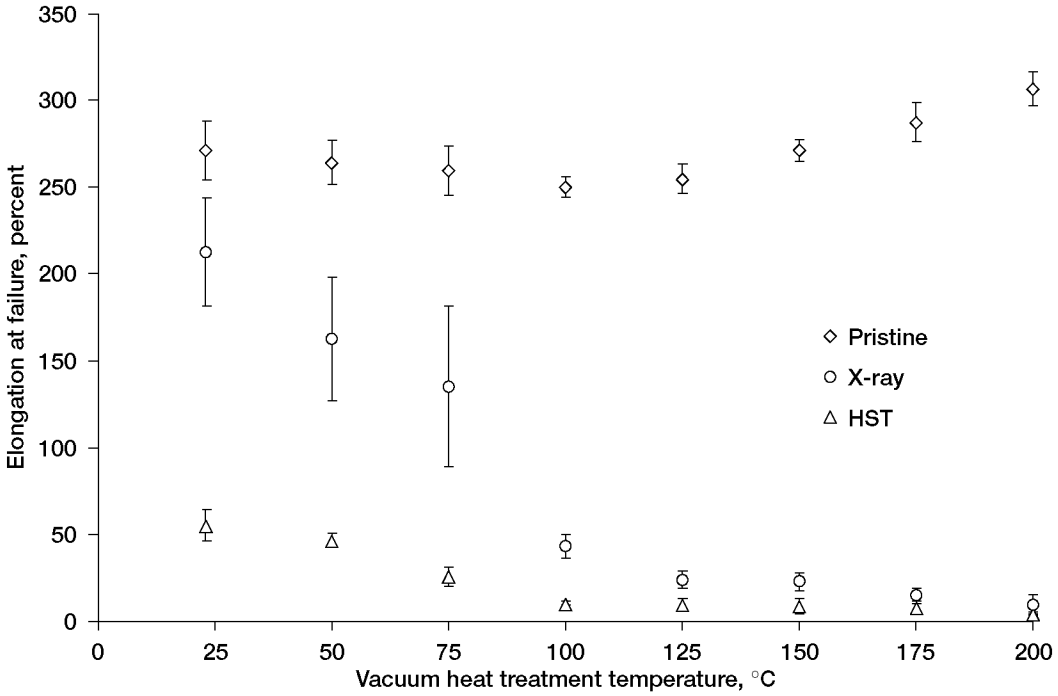

Figure 4. Percent elongation at failure of pristine, ground laboratory x-ray exposed and retrieved HST Teflon FEP as a function of vacuum heat treatment temperature.

5.2.2 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 4. The data are graphed in Figure 5. The standard deviation is given when more than one sample was measured and averaged. 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. This indicates 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 at a particular temperature for the samples that had been irradiated either in space or in the ground facility than for pristine FEP. These results are consistent with de Groh's previous studies that show pristine FEP increases in density with heating, but FEP from HST has greater increases in density for the same heat treatment (200°C exposure, reference 7 and 9). This is attributed to irradiation-induced scission of bonds in space, which allows for greater mobility and crystallization upon heating than that which occurs with non-irradiated FEP. Previous x-ray diffraction studies verify that the increases in density correlate to increases in polymer crystallinity.[7,9] The density results further support chain scission as the primary mechanism of degradation of FEP in the space environment.

Table 4. Density Data of Vacuum Heat-Treated Pristine, X-Ray Irradiated and HST Retrieved Teflon FEP.

Temperature (°C)	Pristine FEP		X-Ray FEP		HST FEP (SM3A 2001 BM-R1)	
	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

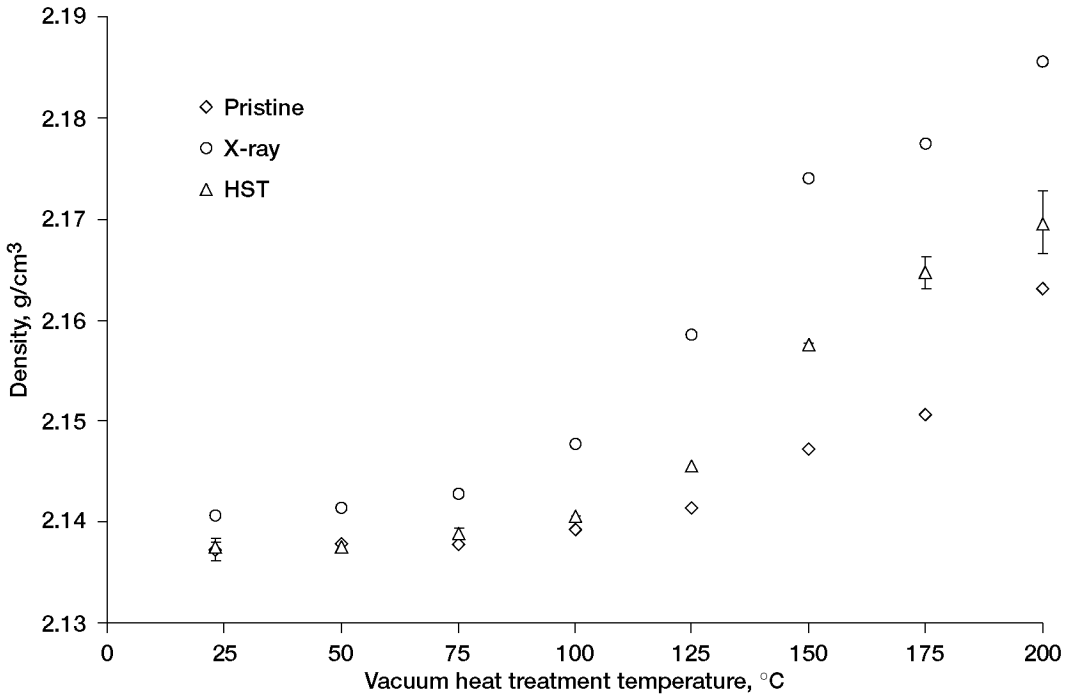

Figure 5. Density of pristine, ground laboratory x-ray exposed and retrieved HST FEP as a function of vacuum heat treatment temperature.

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 was therefore graphed with linear fits for two sections of the data. The 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 Figures 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 I transition temperature (α relaxation), which is listed from ≈ 83°C to 150°C in the literature, dependent on hexafluoropropylene (HFP) content.[9] Eby and Wilson report transition temperatures for FEP with densities (2.136 to 2.135 g/cm^3) similar to the pristine FEP examined in this report at ≈ 150°C and ≈ 127°C for 10.7 and 17.7 mol % HFP, respectively.[18] Commercially available FEP is reported to be 20 mol % HFP,[19] 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 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 increases in crystallization to occur at a lower temperature than which it occurs in pristine FEP.

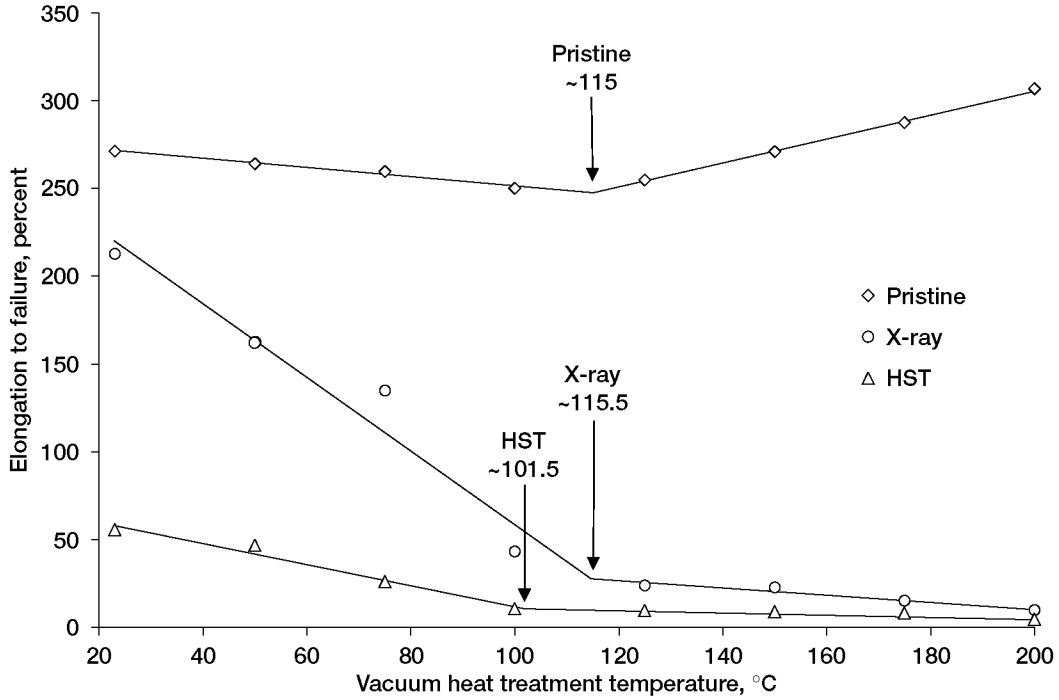

Figure 6. Change in the slope of the percent elongation at failure data of pristine, ground laboratory x-ray exposed and retrieved HST FEP as function of vacuum heat treatment temperature.

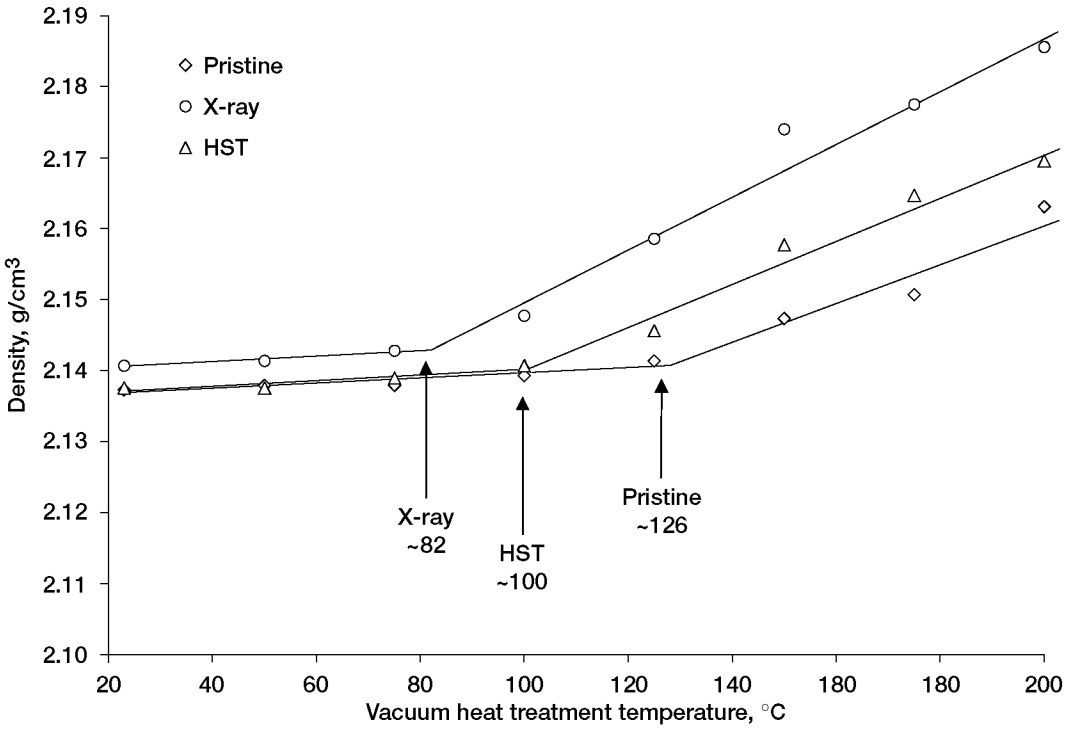

Figure 7. Change in the slope of the density data of pristine, ground laboratory x-ray exposed and retrieved HST FEP as function of vacuum heat treatment temperature.

6.0 Summary and Conclusions

The objective of this research was to determine the effects of heating on ground laboratory irradiated 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 LEO environment. Samples of pristine FEP, x-ray irradiated FEP and HST SM3A-retrieved FEP were heated from 50°C to 200°C in 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 non-irradiated 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 non-heated 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°C 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 non-irradiated 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 I 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 primary 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 in the LEO space environment.

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13. ABSTRACT ***(Maximum 200 words)***

The outer most layer of the multilayer insulation (MLI) blankets on the Hubble Space Telescope (HST) is back surface aluminized Teflon® FEP (fluorinated ethylene propylene). As seen by data collected after each of the three servicing missions and as observed during the second servicing mission (SM2), the FEP has become embrittled in the space environment, leading to degradation of the mechanical properties and severe on-orbit cracking of the FEP. During SM2, a sample of aluminized-FEP was retrieved from HST that had cracked and curled, exposing its aluminum backside to space. Because of the difference in optical properties between FEP and aluminum, this insulation piece reached 200 °C on-orbit, which is significantly higher than the nominal MLI temperature extreme of 50 °C. This piece was more brittle than other retrieved material from the first and third servicing missions (SM1 and SM3A, respectively). Due to this observation and the fact that Teflon thermal shields on the solar array bi-stems were heated on-orbit to 130 °C, experiments have been conducted to determine the effect of heating on the degradation of FEP that has been irradiated in a ground laboratory facility or in space on HST. Teflon FEP samples were x-ray irradiated in a high vacuum facility in order to simulate the damage caused by radiation in the space environment. Samples of pristine FEP, x-ray irradiated FEP and FEP retrieved from the HST during SM3A were heat treated from 50 to 200 °C at 25° intervals in a high vacuum facility and then tensile tested. In addition, samples were tested in a density gradient column to determine the effect of the radiation and heating on the density of FEP. Results indicate that although heating does not degrade the tensile properties of non-irradiated Teflon, there is a significant dependence of the percent elongation at failure of irradiated Teflon as a function of heating temperature. Irradiated Teflon was found to undergo increasing degradation in the elongation at failure as temperature was increased from room temperature to 200 °C. Rate of degradation changes, which were consistent with the glass I transition temperatures for FEP, appeared to be present in both tensile and density data. The results indicate the significance of the on-orbit temperature of Teflon FEP with respect to its degradation in the low Earth orbital space environment.

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