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## **ANALYSIS OF RETRIEVED HUBBLE SPACE TELESCOPE THERMAL CONTROL MATERIALS**

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### **ABSTRACT**

The mechanical and optical properties of the thermal control materials on the Hubble Space Telescope (HST) have degraded over the nearly seven years the telescope has been in orbit. Astronaut observations and photographs from the Second Servicing Mission (SM2) revealed large cracks in the metallized Teflon<sup>®</sup> FEP, the outer layer of the multi-layer insulation (MLI), in many locations around the telescope. Also, the emissivity of the bonded metallized Teflon<sup>®</sup> FEP radiator surfaces of the telescope has increased over time. Samples of the top layer of the MLI and radiator material were retrieved during SM2, and a thorough investigation into the degradation followed in order to determine the primary cause of the damage. Mapping of the cracks on HST and the ground testing showed that thermal cycling with deep-layer damage from electron and proton radiation are necessary to cause the observed embrittlement. Further, strong evidence was found indicating that chain scission (reduced molecular weight) is the dominant form of damage to the metallized Teflon<sup>®</sup> FEP.

**KEY WORDS:** LEO Environmental Effects, Teflon<sup>®</sup> FEP (fluorinated ethylene propylene), Hubble Space Telescope

### **1. INTRODUCTION**

The Hubble Space Telescope was launched into Low Earth Orbit (LEO) in April 1990 with two types of thermal control surfaces: Multi-Layer Insulation (MLI) blankets and bonded radiator surfaces (1). During the First Servicing Mission (SM1) in December 1993 MLI blankets were retrieved and were subsequently analyzed in ground-based facilities. These studies revealed that the outer layer of the MLI, aluminized Teflon<sup>®</sup> FEP (fluorinated ethylene propylene), was beginning to degrade. Close inspection of the Teflon<sup>®</sup> FEP revealed through-thickness cracks in areas with the highest solar exposure and stress concentration. Mechanical tests showed that the ultimate strength and elongation of the Teflon<sup>®</sup> FEP had reduced significantly (2). During the Second Servicing Mission (SM2) in February 1997, astronauts observed and documented severe cracking in the outer layer of the MLI blankets on both solar facing and anti-solar facing surfaces (1). During the repair process, a small specimen of the outer layer was retrieved from the Light Shield (LS) region and was returned for ground-based analysis. In addition, as part of an

instrument installation, a sample of the bonded Teflon® FEP radiator surface was returned on the cryo-vent cover (CVC).

Since the damage to the outer layer was so severe at SM2, a Failure Review Board was convened to, among other tasks, determine the mechanism of the damage. This effort consisted of two major investigations. First, the specimens retrieved during the servicing missions were characterized exhaustively using mechanical and chemical analysis methods to understand the mechanism of the damage. Second, pristine samples of the material were exposed to simulated space environments in an effort to duplicate the condition of the retrieved specimens and determine what element(s) of the space environment caused the damage. Although some of the individual results are detailed elsewhere in this volume (3, 5, 14), this paper summarizes all of the results and draws overall conclusions about the failure mechanism based on those results, the observations of HST, and the calculated fluences.

## 2. MATERIALS

Two types of thermal control materials were investigated. The first type, the MLI blanket, was composed of several underlying layers of aluminized Kapton®, and a top (space-exposed) layer of aluminized Teflon® FEP. The top layer was 127 µm (0.005 in) Teflon® FEP with roughly 100 nm of vapor deposited aluminum (VDA) on the back (FEP/VDA). The layers of the MLI were bonded together at the edges of the blanket assembly with an acrylic adhesive. The bottom layer was attached to the spacecraft with Velcro® (1).

The HST radiator surfaces used the second type of thermal control material. The material consisted of 127 µm (0.005 in) Teflon® FEP with roughly 100 nm of vapor deposited silver (VDS) on the back (FEP/VDS). The silver side was coated with Inconel and then with an acrylic adhesive. The entire sheet was then bonded directly to the spacecraft (1).

MLI blankets were removed from the HST magnetometers during SM1. A nominal specimen, from a region with average solar exposure [11,339 equivalent solar hours (ESH)], was designated MLI SM1 for this investigation. A second MLI specimen was retrieved from the LS area during SM2 and was designated MLI SM2. A sample of the radiator material (FEP/VDS) was retrieved during SM2 on the cryo-vent cover (CVC). This specimen was designated CVC SM2. In addition to the flight samples, a control sample of the MLI was provided by Lockheed Martin Missiles and Space, and was designated "pristine".

In this paper, "MLI specimen" refers to the outer layer only (FEP/VDA or FEP/VDS), not the full MLI blanket. The specimen designations are summarized in Table 1 below.

TABLE 1: SPECIMEN NAMES AND DESCRIPTIONS

Specimen Name	Description
Pristine	MLI from Lockheed Martin (FEP/VDA layer), received 4/15/97
MLI SM1	MLI from magnetometer cover (FEP/VDA layer), retrieved at SM1
MLI SM2	MLI from light shield (FEP/VDA layer), retrieved at SM2
CVC SM2	Radiator FEP/VDS from cryo-vent cover, retrieved at SM2

## 3. ANALYSIS OF RETURNED SPECIMENS

The SM2 flight specimens were fully documented using macro photography, optical microscopy, and scanning electron microscopy (SEM). Then the MLI specimens from SM1 and SM2 were characterized through exhaustive mechanical, optical, and chemical testing.

**3.1 Scanning Electron Microscopy (SEM) and Optical Microscopy** The first task was to document the MLI SM2 specimen and assemble the four pieces received into the single specimen that was cut in orbit. Both SEM and optical microscopy were utilized in this effort. Once the original configuration had been determined, the edges were identified as either a deliberate cut, a handling artifact, or an on-orbit fracture (see Figure 1). From this information, the fracture initiation site became apparent.

The fractures that resulted in the MLI SM2 specimen initiated at an edge of the MLI that had been cut to fit around a handrail. From small defects in this cut edge, two fractures developed and propagated in orbit almost normal to one another, resulting in a roughly triangular specimen. The VDA was completely missing from the MLI SM2 specimen in regions where the Teflon® FEP was bonded to the rest of the blanket, which included the region where the cracks initiated.

Although the blankets were relatively flat when deployed, photos of the MLI SM2 specimen in orbit showed that it was tightly curled, with the space-exposed Teflon® FEP surface as the inner surface and the VDA exposed (1). This curling indicated a volume gradient in the specimen. Based on the diameter of the curl (1.5 cm) the estimated strain difference between the outer and inner surface of the Teflon® FEP was ~1.5% (3).

SEM images of the initiation region show clear differences between the scissors cut that occurred prior to launch or on orbit, the fractures that propagated while in orbit, and cracks from subsequent handling (3; figures 3-8). The featureless nature of the orbital fracture is unique, and attempts to duplicate this smooth fracture with the SM2 specimen under bending or tensile stress resulted in fractures with more fibrous features (3; figures 1, 2, 9). The inability to duplicate the featureless fracture indicated that the fractures propagated in orbit very slowly, in the presence of relatively low stress and under the influence of radiation and other environmental factors. This type of “slow crack growth” has never been studied in Teflon® FEP (3).

Homogeneous mud-like cracking (mud-tiling) and buckling of the VDA were also apparent in the SEM and optical images. A mismatch between the coefficient of thermal expansion (CTE) of the Teflon® FEP and the VDA was most likely the cause (3, 19). Tensile cracks would develop in the aluminum from low cycle fatigue as the material was cycled above room temperature, and buckling would occur when the material was cycled below room temperature (3; figure 17).

The mud-tiling of the metal backing was apparent in all of the specimens. In the CVC specimen, handling and processing procedures such as bending and pressing the FEP/VDS while adhesive bonding it to the spacecraft surface most likely created the cracks. SEM images of the surface show recession and texturing common in polymers exposed to a sweeping ram fluence of atomic oxygen.

**3.2 Mechanical Analyses** Mechanical properties were studied using the following instruments and techniques: Instron Mini Tester, Confocal Microscope, bend testing, and Nano Indenter II Mechanical Properties Microprobe (MPM).

**3.2.1 Instron Mini Tester** The most obvious indication of degradation in the MLI specimens was found in the tensile test results. Reference 3 contains details of the testing and analysis. Table 2 (below) is a summary of the strength test results. In terms of strength, the MLI SM2 specimen was obviously most degraded. The CVC SM2 specimen was less degraded, and the strength of the MLI SM1 specimen degraded the least. This ranking was most apparent in the elongation data.

TABLE 2: SUMMARY OF STRENGTH TEST RESULTS (4)

Material	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)
pristine	13.8	24.8	340
	14.3	26.5	360
	14.3	28.1	390
MLI SM1	14.3	15.4	196
	14.3	16.6	116
CVC SM2	11.0	12.1	25
	15.4	16.0	25
	N/A	11.0	15
MLI SM2	N/A	13.2	0
	N/A	2.2	0

**3.2.2 Confocal Microscope** Thickness measurements were made using a Confocal microscope. Several measurements were made of samples that had been potted and cross sectioned for other analyses. Table 3 (below) gives the thickness measurements of the materials.

TABLE 3: THICKNESS MEASUREMENTS

Material	Thickness ( $\mu\text{m}$ )
pristine	121.4
MLI SM1	120.9 $\pm$ 0.6
MLI SM2 (region 1)	111.2 $\pm$ 0.5
MLI SM2 (region 2)	113.8 $\pm$ 1.4

The MLI SM1 sample was taken from one of the two returned magnetometer covers. This thickness is considerably higher than that reported in reference 2 for a region with comparable solar hours, however, this specimen was from a different magnetometer cover.

**3.2.3 Bend Testing** Because of the condition of the MLI during SM2, astronauts were directed to bend the outer layer on Bay 8 to determine how fragile it was. The Bay 8 specimen, which had not curled, was bent 180 degrees so that the two VDA surfaces touched. Following this, the astronaut found no obvious damage to the material (1). The returned SM2 specimen proved much less durable.

Bend testing was performed on MLI specimens from SM1 and SM2, and on radiator specimens from SM2 (CVC SM2); detailed results are reported in reference 4. Each small specimen was bent manually to 180 degrees around successively smaller mandrels. Following each bend, the specimen was examined with an optical microscope to detect crack length and features. As expected, the pristine material showed no cracking when bent around the smallest mandrel, a strain of 15 percent (4).

Each of the two MLI SM2 samples formed a full-width crack when bent around the first or second large mandrel with the space-exposed surface in tension. Examination showed that this single, full-width crack went most of the way through the thickness of the sample, although the strain from the mandrel diameter was only 2 to 2.5 percent. SEM analysis of the fractures showed the fibrous features of a handling crack. Bending two other MLI SM2 samples around the smallest mandrel with the space-exposed surface in compression did not produce cracks, even at the resulting 15 percent strain. This implied that the space-exposed surface was more brittle than the back surface (4).

The MLI SM1 specimens and the CVC SM2 specimens cracked quite differently from the MLI SM2 specimens. Instead of a single, catastrophic crack, the specimens developed several very short, shallow cracks that eventually joined to form a long, jagged crack across the surface at much smaller mandrels (higher strain). Existing flaws from vent cuts or handling reduced the strain at which cracks first appeared. Unlike the MLI SM2 samples, these samples appeared to retain considerable fracture toughness (4).

**3.2.4 Nano Indenter II Mechanical Properties Microprobe (MPM)** The surface micro-hardness of MLI specimens from SM1 and SM2 and from radiator specimens from SM2 (CVC SM2) were measured by Nano Instruments using their patented Continuous Stiffness Measurement technique. The results are reported in detail in reference 4. All of the space-exposed specimens showed an increased hardness at the surface that decreased with depth. By 500 nm, the hardness of all the exposed specimens was indistinguishable from that of pristine at 500 nm. Although the SM1 materials seemed to show a trend of increasing hardness with increasing solar exposure, the SM2 materials, which had the highest solar exposure, did not follow this trend (4).

**3.3 Optical Analyses** The optical properties were studied using a UV-Vis-NIR Spectrophotometer (Cary 5E, Varian) and a Laboratory Portable Spectroreflectometer (LPSR).

Significant effort was spent in determining the appropriate method for measuring the solar absorptance of the flight materials. Because of the mud tiling and the delamination of the metal

coatings, traditional methods gave results that either over- or under-estimated the changes to the solar absorptance. Reference 5 details the different methods that were considered and the results of the various tests.

For the MLI SM2 specimen, most of the 0.08 solar absorptance increase of the material was attributed to increases in the solar absorptance of the Teflon<sup>®</sup> FEP, rather than to cracking in the VDA. With the VDA removed, the solar absorptance of the MLI SM2 specimen was still 0.06 higher than pristine. No clear correlation was found between solar absorptance increase and equivalent solar hours (ESH).

Literature values for solar absorptance of pristine FEP/VDS were found between 0.06 to 0.09. The increase in the solar absorptance of the CVC SM2 specimen was attributed to darkening of the acrylic adhesive that was used to bond the material to the spacecraft. During the bonding process, the material was repeatedly bent and deformed, which created the mud tiling cracks in the silver deposit. The adhesive bled through these cracks in the silver and was exposed to sunlight. Acrylic adhesives are known to darken when exposed to UV (1, 5).

**3.4 Chemical Analyses** The chemical composition was studied using Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS), Fourier Transform infrared microscopy ( $\mu$ -FTIR), Attenuated Total Reflectance infrared microscopy (ATR/FTIR), and X-ray Photoelectron Spectroscopy (XPS).

**3.4.1 Time-of-Flight Secondary Ion Mass Spectrometry** Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) was used to determine the ion composition of the first mono-layer (0.3 nm) of the specimens and to image ion intensities on the cross sections (6).

For pristine Teflon<sup>®</sup> FEP, the most common fragmentation point was at the bond between  $CF_2$  molecules. Some minor contamination of the surface was found (6).

The spectra of the MLI SM2 specimen had the most evidence of chemical changes. There were many oxygen-containing species, mostly in  $C_x-O_y-F_z$  bonds. In high mass regions of the spectra, C-O-F bonds were more prevalent than C-F bonds. There was some indication of de-fluorination on the surface, but the dominance of the C-O-F bonds weakened the evidence. In the cross section, the highly oxidized ions were present to a depth of 5-10  $\mu m$ . Ion concentrations in the 10-110  $\mu m$  depth were similar to those of the pristine Teflon<sup>®</sup> FEP. Several silicon-containing ions were detected on the surface (6).

The most striking change detected in the CVC SM2 specimen was evidence of de-fluorination on the surface. Ions were detected at intervals that represented only an additional C atom, rather than an additional  $CF_2$  molecule. Although oxygen was detected in a few of the low-mass fragments, the C-F bonds dominated the spectra. Unlike the MLI SM2 specimen, most ions did not contain oxygen. Analysis of the cross section showed a spectrum very similar to pristine Teflon<sup>®</sup> FEP, indicating that the de-fluorinated region was on the very surface of the specimen. Very few silicon-containing contaminants were found on this surface (6).

The spectra of the MLI SM1 specimen most closely resembled the pristine. There was some evidence of oxidation and de-fluorination, but not to the extent present in either of the other two flight specimens. Silicon-containing contaminants were detected on the surface (6).

An in-depth analysis of the contamination was not conducted for this investigation. However, earlier investigations into this type of contamination proved that spacecraft surfaces can be contaminated by silicones in the shuttle bay and solar arrays.

**3.4.2 Fourier Transform Infrared Microscopy** Fourier Transform infrared microscopy ( $\mu$ -FTIR) analysis was performed as described in reference 13, and the details of the analysis can be found in reference 7. The testing conducted for this effort did not confirm that this method can detect crystallinity changes. So, although this method showed no significant differences in the crystallinity of the of the pristine, MLI SM1, MLI SM2 or CVC SM2 specimens, the test was inconclusive. Also, only MLI SM1 showed a significant amount of oxidation in the first 3 to 5  $\mu m$  of the material (7).

**3.4.3 X-ray Photoelectron Spectroscopy** X-ray Photoelectron Spectroscopy (XPS) was performed on the MLI SM2 and CVC SM2 specimens and a pristine specimen. The analysis depth of the XPS is roughly 100 Å. In this case, a change in the ratio of carbon to fluorine (C/F) was defined as damage (8).

The C/F ratio of pristine Teflon® FEP was 8.05 with an oxygen concentration of 0.2 atom%. The CVC SM2 specimen appeared to be the most damaged with a measured C/F ratio of 6.3, and an oxygen concentration of 1.9 atom%. A typical region of the MLI SM2 specimen had a C/F ratio of 6.8 with an oxygen concentration of 0.8 atom%. A region of the MLI SM2 specimen that appeared contaminated was the least damaged with a measured C/F ratio of 7.9, an oxygen concentration of 1.5 atom%, and a trace contaminant of either silicone or hydrocarbon (8)

**3.5 Molecular Structure Analyses** The molecular structure was investigated using X-ray Diffraction (XRD), density gradient column, and Solid-State Nuclear Magnetic Resonance Spectroscopy (NMR).

**3.5.1 X-ray Diffraction** X-ray Diffraction (XRD) was used to detect changes in the crystallinity of the returned MLI specimens from SM1 and SM2. The details of this analysis can be found in reference 9, and the results are summarized in Table 4 (below).

The pristine specimen had a crystallinity of 28-29%. Specimens with various ESH returned during SM1 showed a crystallinity of 28-32%. These measurements were within the uncertainty of the instrument, so MLI SM1 specimens had a crystallinity that was indistinguishable from pristine. The SM2 specimens showed a significant increase, with a crystallinity of 46-47% (9).

**3.5.2 Density Gradient Column** The density of the specimens was found using a density gradient column. This data was then converted to crystallinity values using a table provided by DuPont. This method was outlined in reference 10, and the results are summarized in Table 4 (below).

The calculated crystallinity of the SM1 specimens were indistinguishable from pristine material at 50%. The crystallinity of the MLI SM2 specimen was higher, at 65% (10).

Although there were differences between the absolute value of the crystallinity determined using XRD and the density method, the change in crystallinity is identical. Both methods show an increase in crystallinity of 15%. The different absolute values of the two methods was not surprising because the principles involved were so different. Based on literature data comparing XRD to various methods, a difference in absolute crystallinity of up to 14% is not uncommon (9).

TABLE 4: SUMMARY OF CRYSTALLINITY RESULTS

Sample	ESH	XRD		Density Gradient Column	
		# Tested	Crystallinity (%)	Density (g/cm <sup>3</sup> )	Crystallinity (%)
Pristine 5 mil FEP	0	6	28-29	2.1400	50
Pristine FEP/VDA	0			2.1394	50
MLI SM1	4,477	1	30	2.1375	49
	6,324 or 9,193	1	29	2.1381	50
	9,193 or 6,324	1	32	2.1381	50
	11,339	2	29-30	2.1378	50
	16,670	1	32	2.1406	50
MLI SM2	33,638	2	46-47	2.1836	65

**3.5.3 Solid-State Nuclear Magnetic Resonance Spectroscopy** Solid-State Nuclear Magnetic Resonance (NMR) was performed at the University of Akron on MLI specimens from SM1 and SM2. The results are summarized in reference 10.

NMR performed on the pristine material showed a CF<sub>3</sub> abundance of 7.5%. Analysis of the MLI SM1 specimen detected no significant changes in chemistry or morphology. However, the

analysis of the MLI SM2 specimen showed evidence of changed morphology. The results indicated that the SM2 specimen had undergone chain scission, and that either an increased crystallinity or cross-linking also occurred. No quantitative analysis was feasible.

#### 4. SPACE ENVIRONMENT SIMULATIONS

Several different simulations were employed individually and in combination to determine which elements of the space environment were most likely to cause the damage observed in the returned specimens. Teflon<sup>®</sup> FEP specimens were exposed to combinations of electrons, protons, and thermal cycling at NASA Goddard Space Flight Center (GSFC). Vacuum ultraviolet and soft x-ray radiation exposures were carried out at Brookhaven National Labs (BNL), National Synchrotron Light Source (14).

**4.1 GSFC Radiation and Thermal Cycle Exposures** The initial purpose of the electron and proton radiation exposures carried out at GSFC was to determine the dose at which Teflon<sup>®</sup> FEP would shatter with gentle contact. Specifically, at what servicing mission would the HST MLI outer layer shatter if astronauts tried to remove it or came into contact with it. The approach was to expose specimens of the material to increasing fluences of electrons and protons and then perform tensile tests to determine the changes to the yield and ultimate strengths. When initial testing revealed little change in tensile test data at SM2 fluences, the decision was made to add thermal cycling to the test matrix. The modified test procedure and results are outlined below (11, 12).

**4.1.1 Procedure** Twenty-eight tensile test specimens (ASTM D1822, Type L Die) were cut with identical orientation from a single sheet of Teflon<sup>®</sup> FEP. The GSFC Radiation Effects Task Group exposed a set of three specimens to each of the fluence of electrons and protons listed in Table 5 below. Each fluence was based on the estimated fluence at a specific HST servicing mission.

TABLE 5: FLUENCES FOR GSFC RADIATION EXPOSURES

Run	Protons (1 MeV) $\times 10^{10}/\text{cm}^2$	Electrons (0.5 MeV) $\times 10^{13}/\text{cm}^2$	Equivalent HST Mission	HST Fluence Years	Number of Thermal Cycles ( $\pm 50$ )
1	1.956	1.949	SM2	6.8	-
2	2.771	2.740	SM3	9.6	39,712
3	3.567	4.130	SM4	13.2	56,304
4	5.861	6.040	EOL	20	77,088
5	11.72	12.08	2xEOL	40	116,800
6	29.30	30.20	5xEOL	100	-

Following the irradiation of a set of specimens, one specimen was thermal cycled. The other two were tensile tested to determine the effect of the radiation alone on tensile properties. An unexposed control specimen was tensile tested along with each set to verify the consistence of the test procedure (11).

The temperature limits of the thermal cycling were based on the nominal limits for the MLI outer layer in orbit. Based on the thermal properties of the FEP/VDA, the MLI outer layer of solar-facing surfaces reached +50 °C when in the sun, and dropped to -100 °C when in shadow (1). Although these limits changed when the MLI SM2 specimen curled and exposed the VDA, since most of the damaged surfaces on HST did not curl, these limits were used for the experiment.

Rapid thermal cycling between -100 °C and +60 °C took place in a nitrogen atmosphere in a modified thermal cycle chamber. Liquid nitrogen vapor and a heat gun were added to the chamber to reduce the period of the cycles to 15 seconds. Temperatures were monitored with thermocouples all around the test specimen, and the cycle was driven by a thermocouple affixed with epoxy to a control specimen mounted adjacent to the test specimen (12).

Following thermal cycling, the specimens were tensile tested and changes in tensile properties were noted (11).

**4.1.2 Results** Table 6 below summarizes the results of the combined testing. The loads and elongation can be calculated from the following gauge dimensions: area, 0.127 mm x 318 mm; length, 1.905 cm.

The data indicate that yield strength was unchanged by radiation and slight evidence that yield strength increased due to subsequent thermal cycling. The ultimate strength clearly was reduced by radiation, with roughly 25% loss at the 20 year HST end-of-life (EOL) fluence. Adding thermal cycles reduced the ultimate strength by another 15%. As with the returned SM2 specimens, the changes are most apparent in the elongation data. With the EOL radiation fluence, the elongation had lost 18% of the pristine value. Following thermal cycling, the elongation lost another 28%. Although these EOL fluences were significantly higher than what HST experienced at SM2, the trends in the data compared favorably.

TABLE 6: TENSILE TEST RESULTS FOLLOWING RADIATION AND THERMAL CYCLING (11)

Run	Radiation Fluence (years)	Thermal Cycles	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation at Failure (%)
Control (10 specimens)	0	0	14.2 ± 0.2	25.1 ± 0.3	356 ± 8
1	6.8	0	14.0	23.2	345
		0	14.5	25.9	329
		0	13.9	25.4	377
2	9.6	0	13.5	20.2	314
		0	13.8	21.0	321
		39,000	14.3	17.6	284
3	13.2	0	13.8	19.9	301
		0	13.8	21.5	280
		56,804	14.4	18.0	267
4	20	0	13.8	19.5	301
		0	13.8	19.0	280
		77,088	14.3	15.4	267
5	40	0	13.8	18.2	293
		0	13.8	16.9	263
		116,800	14.9	14.3	132
6	100	0	13.5	14.3	233
		0	13.7	13.4	180

**4.1 Brookhaven National Labs Exposures** Earlier investigations indicated that soft x-rays from solar flares may have caused the degradation observed on SM1 specimens (13). The National Synchrotron Light Source (NSLS) at Brookhaven National Labs (BNL) can provide very high flux radiation in the desired energy with very tight bandwidths. Using two different beamlines, Teflon<sup>®</sup> FEP specimens were exposed to radiation at several energies in the vacuum ultraviolet (VUV) and soft x-ray region of the spectrum in an attempt to confirm the earlier work and determine if there was a region of the spectrum to which Teflon<sup>®</sup> FEP was particularly sensitive. The procedure and results of these experiments are detailed in reference 14 of this volume.

The study found that exposure to synchrotron radiation of narrow energy bands between 69 eV and 1900 eV was capable of causing degradation in the mechanical properties of Teflon<sup>®</sup> FEP. However, Teflon<sup>®</sup> FEP samples exposed to synchrotron radiation of doses significantly greater than HST end-of-life (EOL) doses did not show loss of tensile strength or elongation comparable to that of the severely embrittled SM2 specimens. Based on these results, it was concluded that VUV and soft x-ray radiation alone were not sufficient to cause the severe degradation in mechanical properties observed in Teflon<sup>®</sup> FEP materials exposed to the HST environment for 6.8 years. Some evidence of wavelength-dependence in the damage was observed for samples



exposed to 290 eV synchrotron radiation which is at the carbon absorption edge. These samples showed surface cracking after tensile testing.

## 5. DISCUSSION

**5.1 Analysis of Returned Specimens** The mechanical properties of specimens that were returned from the second servicing mission were significantly degraded. Curling in the MLI SM2 specimen indicated a volume shrinkage gradient through the thickness, and bend test results confirmed that the space-exposed surface was more embrittled than the inside surface. Fractographic examination of the cracks that occurred in orbit indicated that they propagated very slowly under relatively low stress in the presence of radiation or other environmental effects. Similar featureless fracture surfaces were found in the few small cracks in the SM1 specimens as well. This "slow crack growth" phenomenon has never been studied in Teflon<sup>®</sup>.

Crack patterns in the vapor deposited metal coatings on the back of the thermal control materials resembled homogeneous mud cracks. This "mud tiling" can be caused by thermal cycling or handling. When the material was bonded, as with the radiator surfaces on HST, the adhesive bled through these cracks in the metal and darkened in the presence of ultraviolet radiation, causing increases in the solar absorptance.

The chemical analysis techniques used did not yield consistent results. TOF-SIMS data (analysis depth of 0.3 nm) indicated that the MLI SM2 specimen was the most damaged, with oxygen-containing ions dominating the mass spectra. The XPS data (analysis depth of 10 nm) indicated that the SM2 CVC specimen was most changed, with the lowest C/F ratio. Infrared microscopy (analysis depth of 3  $\mu\text{m}$ ) was inconclusive with respect to crystallinity, although some contamination was found on the MLI SM1 specimen. The differences may have been simply a function of the analysis depths and sensitivities of the different techniques. Limited attempts to determine the chemical composition deeper into the bulk of the material with these techniques found no changes at significant depth. Therefore, it is unlikely that composition changes (e.g. de-fluorination, oxidation) can explain the changes to the bulk properties observed in the retrieved specimens.

The reduced elongation in the SM2 specimens, as evidenced by the tensile and bend test results, demonstrated the material's loss of plastic deformation capability. Since plastic deformation is a function of chain entanglements and, thus, chain length, this indicated a reduced molecular weight in the returned Teflon<sup>®</sup> FEP. This implied that chain scission, rather than crosslinking, was the dominant damage mechanism in the SM2 materials (18). Although it was not as pronounced, similar reduction in elongation and ultimate strength occurred in the SM1 materials.

The density measurements and XRD analysis of the MLI SM2 specimen revealed a 15% increase in crystallinity. The NMR analysis confirmed that both chain scission and increased crystallinity occurred in the MLI SM2 specimen and found no change in the bulk molecular structure of the MLI SM1 specimen.

With these analytical results, the condition of the returned specimens was well documented, and the type of damage was well characterized, but the cause of the damage was still not apparent.

**5.2 Space Environment Simulations** Based on the results from the synchrotron radiation exposures, it was clear that neither VUV nor soft X-ray radiation alone could cause the observed bulk damage to the HST thermal control materials. These wavelengths did reduce elongation at extremely high fluences, however, even at doses several orders of magnitude higher than experienced by HST at SM2 there were no comparable bulk property changes (14).

The documentation of the condition of the blankets in various locations around HST during the two servicing missions was revealing. At the first servicing mission, there were very few macroscopic cracks. A few were discovered near the NASA logo on the anti-solar-facing side of the spacecraft, and a few were found on close examination of the returned materials from the solar-facing side. However, in general, the outer layer of the MLI blankets appeared to be intact (1). During SM2, cracks all around HST were visible to astronauts and in photographs. The damage appeared to be worse on the solar-facing side of HST, but the MLI on the anti-solar side

was still significantly damaged. Cracks were prevalent on both sides of the spacecraft; the cracks on the solar-facing side tended to be longer (1).

Note that the anti-solar-facing side of HST only received albedo ESH equivalent to roughly 10 % of the solar-facing ESH (1). This meant that at SM1 the solar-facing surfaces of HST had received five times more ESH than the anti-solar facing surfaces had received at SM2. If any component of the ESH was the dominant damaging environmental factor, the damage to the solar-facing side should have been far worse at SM1 than the anti-solar facing surfaces. It can be argued that the damage to the solar-facing materials should have been worse at SM1 than the damage to anti-solar facing surfaces at SM2. The photographic evidence of HST clearly contradicts this supposition. This, coupled with the BNL results, casts strong doubt on the idea that any component of the solar spectrum, including soft x-rays, could be solely responsible for the damage observed on HST.

Because the damage to HST did not appear to coincide with ESH, components of the space environment that are more closely homogeneous were suspect. The likely candidates were electron and proton fluences and thermal cycling. The GSFC simulations explored the combined effects of radiation and thermal cycling.

The GSFC experiments showed that electron and proton radiation alone affected the tensile properties of the Teflon<sup>®</sup> FEP. The reduced ultimate strength and elongation was apparent at fluences comparable to the HST end-of-life (20 years). Subsequent thermal cycling between -100 and +60 °C reduced these properties further. These particle radiation exposures coupled with thermal cycling produced damage that most closely resembled the HST specimens. However, the study did not duplicate the degree of damage observed on the returned SM2 specimen with SM2 doses of radiation and thermal cycling at nominal limits (-100 to +50 °C).

The MLI SM2 specimen had curled while in orbit, exposing the underlying VDA to the sun. Once the aluminum was exposed, the material cycled from -100 to +200 °C with each 90 minute orbit. Literature data for Teflon<sup>®</sup> FEP showed two second-order transitions, one at -80 °C and another at +80 °C. Cycling through the upper transition temperature could easily affect the nature of the damage. However, since most of the damaged surfaces on HST did not curl, the nominal limits were chosen for the experiment. Further tests are needed to determine the effect of the higher temperature cycling.

Some of the differences between the MLI SM1 specimen and the MLI SM2 specimen could be attributed to the higher temperature cycles of the curled SM2 specimen. Additionally, the astronaut observation following the "bend test" in orbit revealed that a damaged region that did not curl maintained more fracture toughness in orbit than the curled MLI SM2 specimen exhibited in the ground testing. That qualitative difference could be from either the different temperatures experienced by the Bay 8 specimen and the MLI SM2 specimen or from changes that occurred after the SM2 specimen was exposed to atmosphere. Further testing is required to understand the effects of atmosphere on vacuum irradiated Teflon<sup>®</sup> FEP

## 6. CONCLUSIONS

Analysis of the returned specimens showed that both the MLI SM1 and MLI SM2 specimens underwent chain scission. Evidence of increased crystallinity was found only in the MLI SM2 specimen. Solar absorptance changes in the MLI SM2 specimen were attributed to changes in the Teflon<sup>®</sup> FEP and mud tiling in the VDA. Solar absorptance changes in the CVC SM2 specimen were attributed to mud tiling from handling and subsequent darkening of the acrylic adhesive.

The conclusions of the HST MLI FRB were based on the combined evidence of HST damage and data uncovered in ground-based experiments. The FRB concluded the following:

*The observations of HST MLI and ground testing of pristine samples indicate that thermal cycling with deep-layer damage from electron and proton radiation are necessary to cause the observed Teflon<sup>®</sup> FEP embrittlement and the propagation of cracks along stress concentrations. Ground testing and analysis of retrieved MLI*

*indicate that damage increases with the combined total dose of electrons, protons, UV and x-rays along with thermal cycling.*

Tests continue in order to determine the effects of the higher temperature limit the MLI SM2 specimen experienced.

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FIGURE 1: MLI SM2 SPECIMEN WITH CRACKS IDENTIFIED

