DEGRADATION OF TEFLON® FEP FOLLOWING CHARGED PARTICLE RADIATION AND RAPID THERMAL CYCLING

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ABSTRACT

During the Second Servicing Mission (SM2) of the Hubble Space Telescope (HST) severe degradation was observed on the outer layer of the thermal control blankets. Astronaut observations and photographs revealed large cracks in the metallized Teflon® FEP (fluorinated ethylene propylene), the outer layer of the multi-layer insulation (MLI), in many locations around the telescope. In an effort to understand what elements of the space environment might cause such damage, pristine Teflon® FEP was tested for durability to radiation and thermal cycling. Specimens were subjected to electron and proton fluences comparable to those experienced by HST and were subsequently thermal cycled in a custom-built rapid thermal cycle chamber. Tensile tests of the specimens showed that radiation followed by thermal cycling significantly reduced the ultimate strength and elongation of Teflon® FEP.

INTRODUCTION

The Hubble Space Telescope was deployed at an orbital altitude of 598 km (320 nmi) and 28.5° orbit inclination in April 1990. Two types of thermal control materials were used on HST: multi layer insulation (MLI) blankets and bonded radiator surfaces (1). MLI blankets were retrieved during the First Servicing Mission (SM1) in December 1993 and were analyzed in ground-based facilities. The analyses showed that the outer layer of the MLI, aluminized Teflon® FEP, had begun to degrade. When astronauts returned to the telescope in February 1997 for the Second Servicing Mission (SM2), they found severe cracking in the outer layer of the MLI blankets on both solar facing and anti-solar facing surfaces (1). The worst damage was patched, and a small outer-layer MLI specimen from the light shield was retrieved for testing.

The testing of the retrieved specimens following each servicing mission revealed a great deal about the type of damage the FEP sustained. At SM1, close inspection of the outer layer FEP revealed small, through-thickness cracks in regions with the highest solar exposure and stress concentration. Mechanical tests showed that the ultimate strength and elongation had reduced significantly (2). As evidenced by the cracking observed on the telescope MLI, the damage at SM2 was far more severe. By SM2, the material had undergone chain scission sufficient to cause the complete loss of the ability to plastically deform. The elongation had dropped to 0%, and the ultimate tensile strength had dropped by roughly 70%. In addition, fractographic examination of the cracks indicated that they were a type of slow crack growth, which is unusual in polymers (3).

With the testing of the retrieved specimens the type of damage was relatively well understood, however the cause of the damage was unclear. Environmental testing was done to determine what factor of the space environment might cause the observed degradation. Since the MLI damage occurred on all sides of the telescope, environmental factors that were relatively

homogeneous in the HST orbit were suspected. Trapped electrons and protons and thermal cycling were two such environmental factors (3).

Testing was carried out at NASA Goddard Space Flight Center to determine the effects of HST fluences of electrons and protons followed by thermal cycling. The initial purpose of the electron and proton radiation exposures was to determine the dose at which FEP would fragment with gentle contact. Specifically, at what servicing mission would the HST MLI outer layer fragment 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 the 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 in this paper.

EXPERIMENTAL

Materials

The MLI blankets on HST were composed of a top (space exposed) layer of 127 μm (0.005 in) Teflon® FEP with roughly 1000Å of vapor deposited aluminum (VDA) on the back (FEP/VDA) and fifteen underlying layers of 8.5 μm (0.0003 in) embossed, double-aluminized Kapton®. The layers of the MLI were bonded together at the edges of the blanket assembly with an acrylic adhesive. Only the top layer of the blanket, the FEP/VDA, was damaged by the exposure (1). At the time the blankets were built, none of the FEP/VDA was saved for future testing. Therefore, no control material from that production lot was available.

New FEP/VDA was ordered from the blanket shop at Lockheed Martin Missiles and Space in July 1997 and was labeled "pristine". Twenty-eight tensile test specimens (ASTM D1822, Type L Die) were cut from a single sheet of the pristine FEP/VDA for this experiment. The orientation for all of the specimens was identical and parallel to the roll direction. The gauge dimensions of these specimens were: area, 0.127 mm x 3.18 mm; length, 19.05 mm.

Environments

The fluences and doses for the various environmental factors on the HST surfaces are discussed elsewhere in this volume (4). An ideal experiment to simulate the damage observed on the HST would simulate the dose versus depth profile the orbital specimens experienced. However, the energies provided by the source used for this experiment were limited, making it difficult to match the profile. In addition, this experiment was intended to give a conservative estimate of the dose required to make it impossible to handle the material in orbit. Therefore, the decision was made to provide the entire HST fluence of electrons and protons (40 eV to 1 MeV) with 0.5 MeV electrons and 1 MeV protons. These energies provided a dose that was roughly constant through the depth of the specimen, slightly under exposing the front surface of the specimens and slightly over exposing the back surface.

Radiation

The GSFC Radiation Effects Task Group exposed sets of three specimens to each of the fluences of electrons and protons listed in Table 1 (below). Each fluence was based on the estimated fluence at a specific HST servicing mission, with the end-of-life (EOL) defined as 20 years.

TABLE 1: FLUENCES FOR RADIATION AND THERMAL CYCLING

Run	Protons (1 MeV) $x10^{10}/cm^2$	Electrons (0.5 MeV) $x10^{13}/cm^2$	Equivalent Mission	HST Fluence Years	Number of Thermal Cycles (± 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	-

Thermal Cycling

Following irradiation, some of the specimens were thermal cycled. Thermal cycling was carried out by the GSFC Materials Engineering Branch. 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.

The test samples were thermal cycled roughly 40,000 to 117,000 times between +50 and -100 °C. To accomplish this testing in a reasonable amount of time, liquid nitrogen (L N_2) and a hot air gun were used (see Figure 1) to reduce the cycle period to approximately 15 seconds. The samples were cooled to below -100 °C by flowing LN_2 (as well as gaseous nitrogen) over the them. A phase separator attached to the end of the L N_2 inlet produced a L N_2 "mist" that flowed more evenly over the samples. The samples were then heated by use of a hot air gun. The entire setup was located inside a nitrogen-purged thermal chamber, so the hot air gun flowed gaseous nitrogen (N_2) over the samples. This chamber was under constant N_2 purge to prevent moisture from condensing or freezing on the samples. The flow from the hot air gun was reflected off a metal plate onto the samples to diffuse the heat from the gun.

A solid state relay (SSR) was used to open and close a valve that controlled the flow of L N_2. Another SSR was used to turn the hot air gun on and off. A square wave generator was used to toggle the SSRs. When the signal from the generator was one volt, the SSR controlling the L N_2 opened the valve and the SSR controlling the hot air gun was turned off. When the signal from the generator was zero volts, the SSR controlling the L N_2 closed the valve and the SSR controlling the hot air gun was turned on. The signal from the generator was conditioned through two amplifiers (one for each SSR) before reaching the SSRs.

The duty cycle of the square wave was adjusted to achieve the desired thermal cycle. For most of this testing, the L N_2 valve was open about 38 percent of the cycle and the hot air gun was on for the remaining 62 percent of the cycle. The samples were taped and clamped to the test fixture. Several thermocouples were mounted to the test fixture holding the samples and directly to a control sample to monitor temperature and to adjust the duty cycle of the square wave.

Procedure

Tensile test specimens were punched and sent to the Radiation Effects Task Group for electron and proton exposure. Specimens were exposed in sets of three to each of the fluences in Table 1.

Following irradiation, one specimen was thermal cycled. The other two were tensile tested to determine the effect of the radiation alone on the tensile properties. Following thermal cycling, the specimens were tensile tested. An unexposed control specimen was tensile tested along with each set to verify the repeatability of the tensile test procedure. Tensile tests were performed on an Instron 1125 with a 44 N (10 lb) load cell and a strain rate of 2.7 to 6.7 m/m/min.

RESULTS AND ANALYSIS

Data

The yield and ultimate strengths and elongation data are summarized in Table 2 (below). The loads and elongation can be calculated using the gauge dimensions in the Materials section. A typical load versus extension (stress versus strain) curve can be found in Figure 2.

TABLE 2: 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	192
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

Analysis

The data indicate that yield strength was unchanged by the electron and proton radiation (Figure 3). Following irradiation, although the values were consistently lower than the control, most were still within two standard deviations of the control value. Since the sample set was so small, it was impossible to determine if the consistently lower values were indicative of any real change due to the radiation exposure. There was slight evidence that subsequent thermal cycling increased the yield strength. This was particularly evident in exposure set 5 (40 year fluence and 116,800 thermal cycles) with a yield strength of 14.9 MPa; the control value was 14.2 ± 0.2 MPa.

Yield strength represents the ability of a material to deform elastically. Changes to the yield strength could indicate changes in the crystallinity or crosslinking of the polymer, and the crystallinity of FEP is known to increase with elevated temperatures. However, the crystallinity of the specimens could not be measured directly because of the specimen size and the nature of tensile testing.

The ultimate tensile strength (UTS) was significantly reduced following both irradiation and subsequent thermal cycling. At the 20 year HST end-of-life fluence of electrons and protons the ultimate strength had decreased by 23 percent. Following thermal cycling, the ultimate strength had decreased by 39 percent. Although a UTS reduction could not be resolved in the first exposure set (6.8 year fluence, no thermal cycling), the UTS decreased with each subsequent exposure, and thermal cycling always reduced it further (Figure 4). A similar trend was noted in the elongation values.

As with the returned HST specimens, the changes to the bulk FEP were most apparent in the elongation data. With the 20 year EOL fluence of radiation, the elongation had decreased by 18 percent. The additional thermal cycling decreased the elongation by a total of 46 percent. No decrease in elongation was apparent in the first exposure set, however, similar to the UTS, the elongation decreased with each subsequent exposure, and thermal cycling reduced it further (Figure 5).

Elongation measures the material's plastic deformation capability. In polymers, plastic deformation is a function of chain entanglements and chain length. The decreased elongation of these specimens, coupled with the decreased UTS, indicated reduced molecular weight (chain scission). The simplest techniques to measure molecular weight cannot be used with FEP because it is rather inert. As with the crystallinity, it was impossible to measure the molecular weight of these specimens by other techniques due to the sample size and the nature of tensile testing.

Figures 3, 4 and 5 show the tensile properties (yield strength, UTS, and elongation) versus exposure duration. In addition to the values from this experiment, these graphs have the data from the retrieved HST specimens. From these graphs it is clear that the while irradiation and thermal cycling decreases the UTS and elongation, HST-equivalent fluences did not produce the degree of damage that was observed in the retrieved specimens.

The specimen retrieved during SM2 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 (1, 3). Cycling through a much higher temperature limit could easily affect both the nature and the degree 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, however it is likely that cycling through a higher temperature would increase the damage.

It is worth noting that some differences between the damage caused by these exposures and that caused by orbital exposures may have occurred because these exposures were sequential rather than simultaneous. The synergism between various factors in the orbital environment can often produce damage that is very different from what is produced by the individual factors. Although the synergistic damage is often worse than that caused by individual factors, there are a few combinations that actually mitigate the damage. This is another area that requires further investigation before any conclusions can be drawn about whether a simultaneous exposure would increase the damage.

CONCLUSIONS

This experiment showed that electron and proton irradiation alone affected the tensile properties of the Teflon® FEP. The reduced ultimate strength and elongation were apparent at fluences comparable to the HST end-of-life (20 years). Subsequent thermal cycling between -100 and +50 °C reduced these properties further. These particle radiation exposures and thermal cycling produced chain scission in the FEP, damage that resembled the HST retrieved specimens. However, the study did not duplicate the degree of damage observed on the returned SM2 specimens with SM2 fluences of radiation and thermal cycling at the nominal limits.

The HST Multi Layer Insulation Failure Review Board used these data, along with data from other simulations and retrieved specimens to conclude that thermal cycling with deep-layer damage from electron and proton radiation are necessary to cause the observed Teflon® FEP embrittlement and the propagation of cracks along stress concentrations. It is believed that the damage increases with the combined total dose of electrons, protons, UV and x-rays along with thermal cycling (4).

ACKNOWLEDGMENTS

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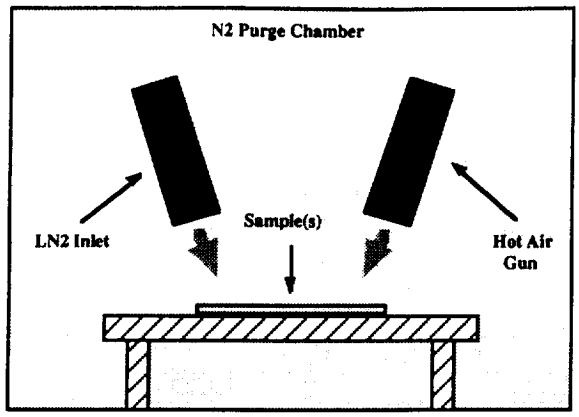

FIGURE 1: SCHEMATIC DIAGRAM OF HIGH-SPEED THERMAL CYCLING SETUP

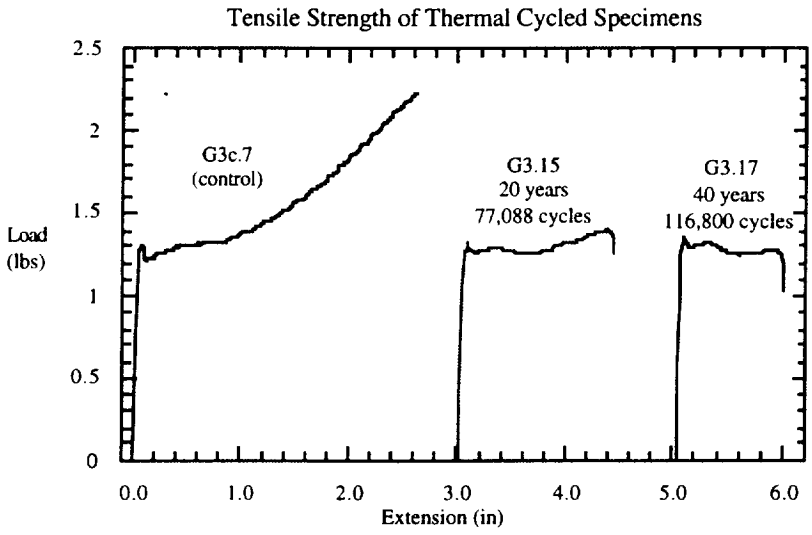

FIGURE 2: TYPICAL TENSILE TEST CURVES - Control Specimen, 20 Year Radiation With 77,088 Thermal Cycles, And 40 Year Radiation With 116,800 Thermal Cycles

FIGURE 3: Yield Strength Versus Exposure Duration

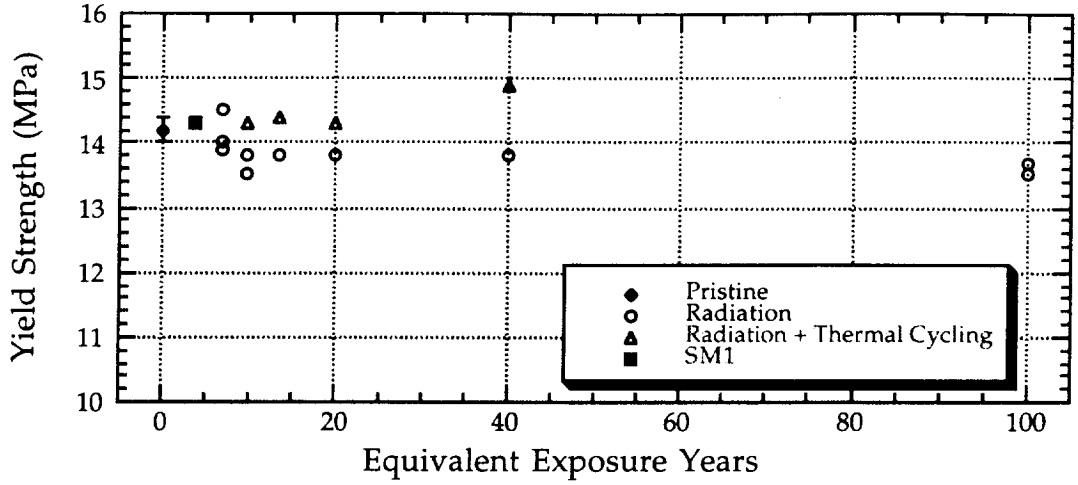

FIGURE 4: Ultimate Strength Versus Exposure Duration

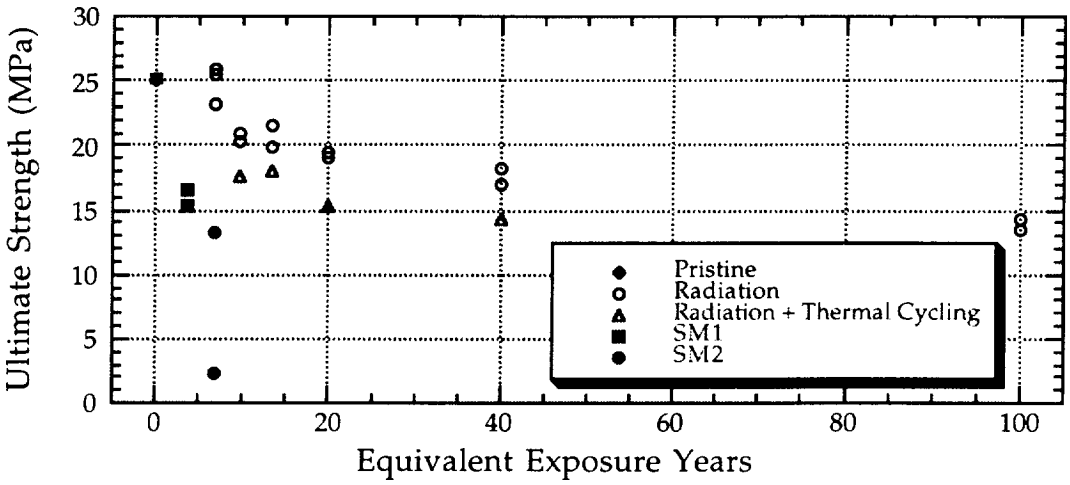

FIGURE 5: Elongation at Failure Versus Exposure Duration