

N 9 3 - 2 8 2 5 9

LDEF POLYMERIC MATERIALS: 10 MONTHS VS. 5.8 YEARS OF EXPOSURE

Philip R. Young
NASA Langley Research Center
Hampton, VA 23681-0001
Phone: 804/864-4265, Fax: 804/864-8312

Wayne S. Slemph
NASA Langley Research Center
Hampton, VA 23681-0001
Phone: 804/864-1334, Fax: 804/864-7729

Alice C. Chang
Lockheed Engineering and Science Company
Hampton, VA 23666
Phone: 804/864-4267, Fax: 804/864-8312

SUMMARY

The chemical characterization of several polymeric materials which received 10 months of exposure and 5.8 years of exposure on a Row 9 Long Duration Exposure Facility (LDEF) experiment (A0134) is reported. Specimens include fluorinated ethylene propylene (FEP) teflon film, polysulfone film and graphite fiber reinforced epoxy and polysulfone matrix composites. The responses of these materials to the two LEO exposures are compared.

The results of infrared, thermal, x-ray photoelectron, and scanning electron microscope analyses are reported. Solution property measurements of various molecular weight parameters are presented for the thermoplastic polysulfone materials. Molecular level effects attributable to exposure that were present in 10-month exposed specimens were not found in 5.8-year exposed specimens. This result suggests that increased atomic oxygen fluence toward the end of the LDEF mission may have eroded away selected environmentally induced changes in surface chemistry for 5.8-year exposure specimens.

INTRODUCTION

The NASA Long Duration Exposure Facility (LDEF) was retrieved from low Earth orbit (LEO) by the Space Shuttle Orbiter Columbia on January 10, 1990. Shortly after that event, the most detailed analysis began of space-exposed materials in the history of the U.S. Space Program. The knowledge being derived from experiments and specimens which spent 5.8 years on LDEF during its 34,000 orbit/three-quarters of a billion mile flight will be the baseline for environmental effects on materials well into the 21st Century. This paper reports the chemical characterization of two sets of polymeric materials which received 10-month and 5.8-year exposures on this remarkable space vehicle.

Previous research in this laboratory dealt with a broad assessment of a variety of polymer films (1-3) and polymer matrix resin composite materials (4,5) on two Langley

Research Center LDEF experiments. With the completion of that assessment came the opportunity to examine individual polymers and phenomena in greater detail.

Materials in the present study are unique because some specimens were flown inside an Experiment Exposure Control Canister (EECC). This canister was closed when LDEF was launched. The EECC opened as programmed 1 month after deployment and closed 10 months later. Specimens of similar materials were placed outside the canister and, thus, received the full 5.8-year exposure. Table I gives environmental exposure conditions pertinent to these two sets of samples. The analysis of these 10-month materials in the EECC is enabling some interesting comparisons with materials which received full exposure.

Figure 1 gives a preflight photograph of Langley's LDEF materials experiment tray. The tray was integrated onto the LDEF structure on Row 9 and Tray B and flew nominally in the RAM direction on the leading edge (6). Recent LDEF supporting data analyses have determined that the actual RAM direction was 8° of yaw from the perpendicular to Row 9, in the direction of Row 10. This tray contained two materials experiments, one dealing with composites (7), and one dealing with coatings and films (8). Most composite specimens are located on the right side of the panel in the photograph. A matching set of pre-cut flight control specimens flew underneath that panel and, thus, were protected from direct exposure. Additional coatings, films, and smaller composite specimens are shown in the center of the tray. The EECC is shown in the open position on the left side of the experiment tray. Many specimens on the experiment tray were held in place by an aluminum template with machined 0.81-inch and 1.35-inch exposure holes.

The detailed chemical characterization of FEP Teflon film, polysulfone film, and graphite fiber reinforced epoxy and polysulfone matrix composites is examined in this report. This characterization included ultraviolet-visible and infrared spectroscopic analyses, thermal analyses, x-ray photoelectron spectroscopy, scanning electron microscopy, and selected solution property measurements of various molecular weight parameters. The intent of this study is to add to the body of knowledge of space environmental effects on materials being derived from the LDEF mission.

EXPERIMENTAL

A description of film and composite materials is given in Table II. Udel P1700 polysulfone film was fabricated in-house from dried resin pellets by applying pressure to a mold heated to 250-300°C and maintaining that pressure for 1 hour before cooling. Other films were obtained from commercial sources or synthesized at Virginia Tech. (9). The fabrication, quality control, specimen preparation, and baseline testing of the composite materials were covered in previous reports (10,11). In general, composite specimens were cut from larger panels processed at the Langley Research Center using prepreg manufacturers' specifications. Matching sets of specimens remained at Langley in a low humidity environment as controls.

Chemical Characterization. The equipment and techniques used to make solution property measurements have been previously reported (12). Gel Permeation Chromatography (GPC) was performed on a Waters Associates System in chloroform using a 10⁶/10⁵/10⁴/10³ Å Microstyrigel column bank. The chromatograph was interfaced with a Viscotek (Viscotek Corp., Porter, TX) Model 100 Differential Viscometer (DV). Thermal analyses were conducted using a DuPont 9900 Thermal Analyzer to process data from DuPont's Model 910 Differential Scanning Calorimeter (DSC) or Model 943 Thermomechanical Analyzer (TMA). Infrared spectra were recorded on a Nicolet 60SX Fourier Transform Infrared System (FTIR) either in

the transmission mode or using a diffuse reflectance (DR) technique (13). Ultraviolet-Visible (UV-VIS) spectra were scanned on a Perkin-Elmer Lambda 4A Spectrophotometer.

A Cambridge StereoScan 150 (Cambridge Instruments, Deerfield, IL) was used for Scanning Electron Microscopy (SEM). An EDAX S150 detecting unit (EDAX International Inc., Prairie View, IL) on the SEM was used to perform Energy Dispersive Spectroscopy (EDS) analyses. The visual appearance of selected specimens was documented using various photographic techniques.

X-ray Photoelectron Spectroscopy (XPS) measurements were conducted at the Virginia Tech Surface Analysis Laboratory (14). Measurements were made on a Perkin-Elmer PHI 5300 Spectrometer equipped with a Mg K α source (1253.6 eV) operating at 15kV/120mA.

DISCUSSION

The chemical and physical response of selected epoxy and polysulfone matrix/graphite fiber reinforced composite materials to 5.8 years of Row 9 exposure has been discussed in previous reports (3-5). The performance of silvered FEP Teflon thermal blanket material after 5.8 years of exposure at various locations has also been the subject of numerous investigations (2,15). The comparison of these materials with identical materials which received only the 10-month, Row 9 exposure provided by the EECC is enabling complementary performance information to be developed.

Polymer Films

FEP Teflon. The only film which survived both the 10-month EECC exposure and the 5.8-year experiment tray exposure was 5 mil thick FEP Teflon. Other thinner, 1 mil thick polymeric films flown outside the canister on this Row 9 experiment failed to survive. Silvered FEP material was flown in both locations; FEP film was only flown inside the EECC. Inspection of FEP and Ag/FEP canister specimens showed no visible effects of exposure. The frosted or diffuse appearance of Ag/FEP after 5.8 years is well documented (2,15).

Figure 2 is the UV-VIS transmission spectrum of the 10-month FEP film. The spectrum was superimposable over that for the control specimen. Figure 3 gives total reflectance curves for Ag/FEP specimens for both exposures. The solar absorptance, α_s , changed only marginally after 5.8 years. Greater detail on the optical and thermal properties of this material may be found elsewhere in this publication (16).

In an earlier study, a small new band near 1730cm⁻¹ was found by subtractive FTIR techniques in the 5.8-year Ag/FEP specimen (2,3). In that study, no other significant differences were noted by FTIR or DSC as a result of exposure. This small 1730cm⁻¹ band was also found in the 10-month specimen by subtracting the FTIR spectrum of the exposed specimen from that for the control. This band is most likely due to the formation of surface carbonyl groups as the result of exposure to the LEO environment. The carbonyl in the infrared spectrum correlated with a 0.5% atomic concentration of oxygen found in this specimen by XPS. No additional molecular level changes were noted with this 10-month specimen. Thus, except for minor changes in surface chemistry, this material was remarkably stable during 10 months of exposure.

Additional Canister Films. In contrast to FEP Teflon film, which showed no visible effects of exposure, other canister films exhibited effects of 10 months in LEO. Figure 4 gives photographs of P1700, Kapton, Kynar, and PIPSX films. The first three films are polysulfone, polyimide, and fluorinated hydrocarbon materials, respectively. PIPSX is an experimental polyimide-polysiloxane block copolymer (9). The exposed area for all four films are apparent in the photograph, as are template-protected areas around their edges. Figure 5 gives UV-VIS transmission spectra for the four materials. The decrease in transmission with exposure is primarily due to AO and UV. Except for P1700 and PIPSX films, no significant molecular level changes were noted by FTIR or DSC. The siloxane copolymer apparently formed a silica/silicate surface layer attributable to AO exposure (1). The performance of a series of PIPSX copolymers flown inside the EECC will be the subject of a future publication.

934/T300 Epoxy Composites

Figure 6 shows the SEM of T300 carbon fiber reinforced 934 epoxy matrix specimens for the two exposures. The 10-month and 5.8-year samples were placed adjacent to each other to enable simultaneous analyses. The left side low magnification photomicrograph shows both exposed surfaces, and surfaces protected from direct exposure by the aluminum retaining template. The fabric-appearing pattern visible in the micrograph was transferred to the composite surface by a glass cloth peel-ply during processing. The right side photomicrograph shows a higher magnification SEM of exposed regions of two specimens. Individual carbon fibers apparent with the 10-month composite are no longer distinguishable after 5.8 years.

Table III gives XPS data for control, 10-month, and 5.8-year epoxy composites. Surface carbon content increased in the first 10 months of exposure. This probably reflects increased carbon fiber content due to preferential erosion of matrix resin. Oxygen and sulfur did not appear to change significantly. Fluorine on the control likely resulted from release agent used during processing. Fluorine was not detected on exposed composites because this outer surface was eroded away by AO. The increased silicon content with exposure is no doubt due to the well-documented LDEF contamination. Additional chemical characterization including FTIR, TMA, and DSC failed to detect significant differences between the two specimens.

The origin and composition of the "white ash" on selected composite surfaces has become one of the mysteries associated with materials flown on LDEF (17). This ash on the 5.8-year 934/T300 epoxy composite was investigated in some detail. Figure 7 shows high magnification SEM photomicrographs of that specimen. Projections rising from the composite surface were apparently caused by contamination protecting underlying material from attack by atomic oxygen. The right hand photograph, obtained by overlaying three individual micrographs, shows graphite fiber fragments presumably sheared off by AO. The white ash in question is visible at the base of this finger-like projection.

Figure 8 shows the ash at higher resolution. The residue appears to contain crystals on the order of 0.1 μ m in diameter. EDS analysis on these crystals, given in Figure 9, revealed sulfur to be a major component. This result was not expected. However, sulfur is present in the diaminodiphenylsulfone (DDS) cured epoxy matrix resin. Similar-appearing residues have been noted on DDS-cured 5208 epoxy and polysulfone composites. The exact chemical composition of this sulfur-containing species has not been established; sodium may be a counter ion. XPS data in Table III shows no unusual sulfur content for the 5.8-year specimen. Apparently, this analysis was not conducted on an ash-rich portion of the exposed composite. Future XPS analyses will focus on this portion (14).

The 934/T300 epoxy composites addressed in this paper were uncoated materials. Thus, they were attacked by the harsh LEO environment. Identical specimens protected with thin coatings, such as 1000Å of nickel followed by 600Å of silicon dioxide, exhibited outstanding resistance to surface erosion. Several inorganic coatings were found to be effective in preventing surface degradation (16).

Polysulfone Film and Composites

Pertinent information on LEO space environmental effects was also obtained by comparing the performance of polysulfone materials after 10 months of exposure with performance after 5.8 years of exposure. Figure 10 shows photographs of 10-month exposed film and 5.8-year exposed composite specimens. Dramatic visual effects in these specimens were primarily due to AO. Protected and exposed surfaces are easily distinguishable in Figure 10.

10-Month Exposed Polysulfone Film. Typical SEM photomicrographs of the polysulfone film are shown in Figure 11. The imprint from scratches on the mold is apparent on the surface of the control film, as are small surface impurities. This contamination, transferred during molding, apparently protected parts of the exposed specimen as evidenced by relief patterns present in that micrograph. AO erosion after 10 months was severe.

UV-VIS spectra of control and exposed film are included in Figure 5. Much of the decrease in transmittance is presumed to be due to UV degradation and AO roughening or "frosting" of the film surface. The glass transition temperature, T_g , of the film apparently was not affected by exposure. Essentially identical glass transition temperatures for control and exposed film were determined by DSC. The T_g of the two specimens determined by TMA and shown in Figure 12 are also identical. No significant change in T_g of any polymer flown on or near the RAM direction of LDEF has been found that could be attributed to LEO exposure.

In contrast to various thermal analyses which detected no difference between control and exposed specimens, FTIR characterization has shown interpretable differences between the two specimens. Figure 13 shows the transmission spectrum of a thin polysulfone film obtained in our laboratory under ideal conditions. Several band assignments have been made in the figure. Since the LDEF specimen was too thick for good quality transmission studies, somewhat poorer quality spectra of control and exposed specimens were obtained by diffuse reflectance (DR). Differences between the two diffuse reflectance spectra were difficult to establish until they were subtracted. The spectrum in Figure 14 is the result of subtracting the DR-FTIR spectrum of the exposed film from that of the control. A downward inflection in the curve is indicative of a larger amount of a particular species in the exposed spectrum.

The band centered around 3400cm^{-1} is most likely due to -OH. Bands at 1485 and 1237cm^{-1} may also be associated with that group. Reports in the literature have noted the 3400cm^{-1} -OH band for polysulfone film exposed to UV (18) and also to 3-MeV protons (19). Additionally, the loss of the 1385cm^{-1} methyl band was noted in at least one study (18). Methyl does not appear to have been lost in the present study. A diminished -CH₃ content would have resulted in an upward inflection in the subtraction spectrum at 1385cm^{-1} ; no band is present around 1385cm^{-1} in Figure 14.

The presence of -OH has been explained by cleavage of the ether oxygen in the backbone of the polymer followed by abstraction of a proton (19), or by a photo-Claisen rearrangement of

the ether oxygen to produce an ortho-hydroxy substituted biphenyl linkage (20). This study made no determination of the origin of the -OH group.

Solution property measurements also revealed molecular level effects of the 10-month LEO exposure. Figure 15 shows the molecular weight distributions of three polysulfone specimens as determined by GPC-DV. Curves for control film, a template-protected specimen cut from around the yellowed edge of the sample, and a directly exposed center-cut specimen are given in the figure. Several points should be made. The solubility decreased from 100% to 96% to 87% for the three specimens. Note also the decrease in number average molecular weight (M_n) and the increase in weight- and z-average molecular weights (M_w and M_z) with exposure. This behavior is considered evidence for both chain scission and crosslinking, thus confirming predictions of ground-based simulation of space environment effects on this material (19-24).

10-Month Exposed Polysulfone Composite. The visual effects of 10 months of exposure of the polysulfone composite specimens were evident, but not as dramatic as those observed for similar specimens exposed for 5.8 years. Figure 16 shows the SEM of a 10-month specimen taken at 4250X magnification. While surface erosion is apparent, individual fibers can still be distinguished. TMA measurements of T_g for control and exposed samples, shown in Figure 12, suggest no change as a result of exposure. DSC measurement of T_g on composites in this study were inconclusive. Subtractive FTIR measurements on 10-month composite specimens showed the same behavior observed for 10-month film. The subtraction composite spectrum is included in Figure 17. The spectrum for the 10-month exposed film is repeated in this figure for comparison purposes. The decreased intensity of the 3400cm^{-1} -OH band for the 10-month exposed composite may be due in part to surface resin content dilution by graphite fiber which probably eroded more slowly than the resin. Solution property measurements on 10-month composite specimens show the general behavior observed on film and given in Figure 15 but less dramatically.

5.8-Year Exposed Polysulfone Composites. As illustrated in Figure 10, 5.8 years of exposure led to severe erosion. Almost one ply of the 4-ply composite was lost. Figure 16b shows the SEM of this material. Both resin and fiber degradation are apparent. The "spider-web"-like residue in the micrograph has also been observed with thermoset composites on LDEF and on Kapton film from the space end of the vehicle (3).

TMA determination of T_g of the 5.8-year specimen, shown in Figure 12, did not detect a change attributable to exposure. An earlier more detailed TMA study of the same material also failed to show a change in T_g (4). Figure 12 illustrates the need to use correct controls when analyzing test specimens. While the three materials were processed at the same time, there were apparently sufficient differences in the cure cycles to produce three different glass transition temperatures. The use of the tensile specimen as a control for the 10-month specimen could have led to an erroneous conclusion.

The 5.8-year composite subtraction spectrum given in Figure 17 is essentially a straight line. This result suggests very little difference in infrared spectra of control and 5.8-year exposed composites. A similar y-axis sensitivity was used to obtain the three spectra in the figure.

An earlier study reported molecular weight determinations made on 5.8-year exposed composite specimens (4). Two pertinent pieces of information from that study, the molecular weight distributions of unexposed resin and ground control, flight control, and flight exposed composites, along with a table of molecular weight values, are reproduced for convenience in Figure 18 and Table IV. The molecular weight distributions were separated for clarity when

Figure 18 was drawn. The distributions are virtually superimposable. Expected variations between techniques are noted for absolute molecular weight values in Table IV. However, a careful inspection of data for the same technique suggests no significant differences in various molecular weight parameters.

The XPS analysis of all specimens in this study is given in Table V. The release agent used during fabrication is apparently present on the control composite surface as evidenced by the 4.1% atomic concentration of fluorine for that sample. Calcium and aluminum were also detected. Silicon, the common LDEF contaminant, is also present with most samples, particularly the 5.8-year exposed composite. The oxygen content for that specimen is unusually high due to the likely formation of silica/silicate upon exposure of the contaminant to atomic oxygen (1, 2). These artifacts make a consistent interpretation of data in Table V difficult.

A Perspective. Evidence developed in this study suggests that molecular level effects present in specimens after 10 months of LEO exposure are not present after 5.8 years of exposure. This potentially contradictory observation is best understood by considering the orbit of the spacecraft during its flight. LDEF was deployed in an essentially circular orbit of 257 nautical miles on April 7, 1984 (25). It was retrieved 69 months later at an altitude of 179 nautical miles. Only about 2 months of orbit lifetime remained at retrieval. The atomic oxygen fluence differs greatly at these two altitudes.

Figure 19 is the approximate cumulative percent RAM AO fluence as a function of time. Exact AO exposure for these specimens is given in reference 26. The 10-month specimens were exposed early in the mission when AO fluence was at a minimum. The 5.8-year specimens received significant exposure near the end of the mission. As much as 50% of total AO exposure was received during the last 6 months in orbit. The molecular level effects observed after 10 months, primarily related to changes in surface chemistry, had most likely been eroded away by the time the satellite was retrieved. An earlier retrieval from a higher orbit may have provided different results.

A second point is also offered. The polysulfone film in this study received 10 months of LEO exposure. Subtle differences at the molecular level, most notably, infrared spectra and solution properties, have been documented. Can this information be used as a benchmark to calibrate ground-based simulation of LEO space environmental effects? If effects on materials described in this report can be simulated, then can the same conditions be used to simulate the effects of LEO exposure on other polymeric materials under consideration for space application? If this is possible, synergistic and accelerated effects may also be better understood.

CONCLUDING REMARKS

The LDEF is providing a wealth of information on the effects of exposure of polymeric materials to the LEO environment. The present study examined the response of several specimens to both 10 months and 5.8 years of RAM exposure on the vehicle. AO induced surface erosion was apparent in both film and composite specimens. Changes were detected in the UV-VIS and IR spectra of some materials after 10 months in LEO, as well as shifts in various molecular weight parameters. Those molecular level effects were not as apparent after 5.8 years of exposure. Rapid surface erosion due to increased exposure to AO toward the end of the mission probably erased some of these fundamental effects. The chemical characterization of additional LDEF-exposed polymeric materials is continuing. The ultimate benefits will be increased confidence in models for spacecraft materials performance in LEO and in better ground-based simulation of LEO space environmental effects on materials.

ACKNOWLEDGMENT

This report was not possible without the capable assistance of a number of individuals. The authors thank Carol R. Kalil, Emilie J. Siochi, Judith R. J. Davis, Karen S. Whitley, and Reta R. Barrett.

REFERENCES

1. P. R. Young, W. S. Slep, and C. R. Kalil; SAMPE Intl. Symp., **37**, 159(1992).
2. P. R. Young and W. S. Slep; NASA TM 104096, December 1991.
3. P. R. Young and W. S. Slep; NASA CP-3134, Part 2, 687(1991).
4. P. R. Young, W. S. Slep, W. G. Witte, and J. Y. Shen; SAMPE Intl. Symp., **36**(1), 40(1991).
5. W. S. Slep, P. R. Young, W. G. Witte, and J. Y. Shen; NASA CP-3134, Part 3, 1149(1991).
6. L. G. Clark, W. H. Kinard, D. J. Carter, and J. L. Jones, Eds.; The Long Duration Exposure Facility (LDEF). NASA SP-473, 1984.
7. W. S. Slep; NASA SP-473, 24(1984).
8. W. S. Slep; NASA SP-473, 54(1984).
9. NASA Grant NAG-1-343, Virginia Polytechnic Institute and State University, Blacksburg, VA.
10. W. G. Witte, Jr.; NASA TM 87624, 1985.
11. W. G. Witte, Jr.; NASA TM 89069, 1987.
12. P. R. Young, J. R. J. Davis, and A. C. Chang; SAMPE Intl. Symp., **34**(2), 1450(1989).
13. P. R. Young, B. A. Stein, and A. C. Chang; SAMPE Intl. Symp., **28**, 824(1983).
14. NASA Grant NAG-1-1186, Virginia Polytechnic Institute and State University, Blacksburg, VA.
15. A. S. Levine, Ed.; NASA CP-3134, Parts 1, 2, and 3. LDEF-69 Months in Space, First Post Retrieval Symposium, Kissimmee, FL, June 2-8, 1991.
16. W. S. Slep and P. R. Young, LDEF Thermal Control Coatings Post Flight Analysis. Presented at the Second LDEF Post-Retrieval Symposium, San Diego, CA, June 1992.
17. B. A. Stein and P. R. Young, Compilers; LDEF Materials Workshop '91, NASA CP-3962, 1992.

18. B. D. Gesner and P. G. Kelleher; J. Appl. Polym. Sci., 12, 1199(1968).
19. D. R. Coulter, M. V. Smith, F. Tsay and A. Gupta; J. Appl. Polym. Sci., 30, 1753(1985).
20. S. Kuroda, A. Nagura, K. Horie, and I. Mita; Eur. Polym. J., 25(6), 621(1989).
21. J. R. Brown and J. H. O'Donnell; J. Appl. Polym. Sci., 19, 405(1975).
22. J. R. Brown and J. H. O'Donnell; J. Appl. Polym. Sci., 23, 2763(1979).
23. K. B. Kingsbury, D. S. Hawkins, R. A. Orwoll, R. L. Kiefer, S. A. T. Long, and G. F. Sykes; Macromolecules, 22, 3486(1989).
24. B. Santos and G. F. Sykes; SAMPE Tech. Conf., 13, 256(1981).
25. R. L. O'Neal and E. B. Lightner; NASA CP-3134, Part 1, 3 (1991).
26. R. J. Bourassa and J. R. Gillis; Atomic Oxygen Exposure of LDEF Experiment Trays, NASA Contractor Report 189627, Contracts NAS1-18224 and NAS1-19247, May 1992.

TABLE I. ENVIRONMENTAL EXPOSURE PARAMETERS

	<u>10-MONTH SPECIMENS</u>	<u>5.8-YEAR SPECIMENS</u>
• ATOMIC OXYGEN	2.6 x 10 ²⁰ atoms/cm ²	8.7 x 10 ²¹ atoms/cm ²
• UV RADIATION 100-400 nm	1,600 hours	11,100 hours
• THERMAL CYCLES	~4,900 -20 to 140°F (±20°)	~34,000 -20 to 160°F (±20°)

Integrated Tray Parameters

• PARTICULATE RADIATION e ⁻ and p ⁺ : 2.5 x 10 ⁵ rad fluence cosmic: <10 rad	• MICROMETEOROID AND DEBRIS 734 impact craters <0.5 mm 74 impact craters >0.5 mm
• VACUUM 10 ⁻⁶ -10 ⁻⁷ torr	• ALTITUDE/ORBITAL INCLINATION 255-180 nautical miles/28.5°

TABLE II. MATERIALS

<u>10-MONTH AND 5.8-YEAR SPECIMENS</u>	<u>EXPOSED THICKNESS</u>	<u>EXPOSED DIAMETER</u>
SILVERED FEP TEFLON	5 mil	0.81 and 1.35 in
934/T300 EPOXY COMPOSITE	4 ply ^a	0.81 in
P1700/C6000 POLYSULFONE COMPOSITE	4 ply ^a	0.81 in and tensile specimen (0.024 in x 0.50 in x 8.0 in)
<u>10-MONTH SPECIMENS</u>		
FEP TEFLON FILM	5 mil	0.81 in
P1700 POLYSULFONE FILM	18 mil	0.81 in
KAPTON FILM	1.2 mil	0.81 in
KYNAR FILM	2.4 mil	0.81 in
POLYIMIDE-POLYSILOXANE COPOLYMER FILM	1.0 mil	0.81 in

^a ~5 mil per ply.

TABLE III. XPS ANALYSIS OF 934/T300 COMPOSITES

Photopeak	Control	10-Month Exposed	5.8-Year Exposed
C 1s B.E. ^a (eV)	285.0 ... 292.3 ^c	283.6 ... 289.7	283.9 ... 288.5
A.C. ^b (%)	68.9	73.3	72.1
O 1s B.E. (eV)	531.5/532.7/533.9	531.3 ... 534.0	531.1/532.5/534.8
A.C. (%)	18.1	18.8	19.7
S 2p B.E. (eV)	168.4	168.6	170.0
A.C. (%)	1.1	0.8	0.9
N 1s B.E. (eV)	399.9	399.6	400.6
A.C. (%)	3.4	5.5	0.8
Si 2p B.E. (eV)	103.2	103.7	104.0
A.C. (%)	1.0	0.9	6.4
Na 1s B.E. (eV)	1072.2	—	—
A.C. (%)	2.0	NSP ^d	—
F 1s B.E. (eV)	689.3	—	—
A.C. (%)	5.5	NSP	NSP

^a Binding Energy.

^b Atomic Concentration.

^c Multiple Peaks.

^d No Significant Peak.

TABLE IV. MOLECULAR WEIGHT OF POLYSULFONE MATRIX RESIN

Sample, Technique	^a M _N	M _w	M _z	M _w /M _N
<u>Resin control</u>				
GPC	19,000	70,000	117,000	3.8
GPC-DV	18,000	50,000	83,000	2.8
GPC-LALLS	21,000	46,000	74,000	2.2
Static LALLS		45,900		
<u>Langley control</u>				
GPC	18,000	68,000	113,000	3.9
GPC-DV	16,000	50,000	81,000	3.1
GPC-LALLS	18,000	40,000	65,000	2.1
<u>Flight protected</u>				
GPC	19,000	68,000	114,000	3.7
GPC-DV	17,000	53,000	87,000	2.9
GPC-LALLS	21,000	40,000	66,000	1.8
<u>Flight exposed</u>				
GPC	18,000	68,000	114,000	3.7
GPC-DV	17,000	48,000	80,000	2.9
GPC-LALLS	21,000	46,000	71,000	2.2

^a Molecular weight in g/mole

TABLE V. XPS ANALYSIS OF POLYSULFONE SPECIMENS

Photopeak	Film		P1700/C6000 Composite		
	Control	10-Month Exposed	Control	10-Month Exposed	5.8-Year Exposed
C 1s B.E. ^a (eV)	285.0/286.6/288.9	283.5 ... 288.7 ^c	283.6 ... 288.9	283.6 ... 288.9	282.0 ... 293.0
A.C. ^b (%)	82.2	79.2	75.5	66.5	25.0
O 1s B.E. (eV)	532.1/533.2/534.3	528.3 ... 534.9	530.7/532.0/533.2	530.3 ... 534.5	530.2 ... 534.5
A.C. (%)	14.3	18.6	14.6	12.4	52.0
S 2p B.E. (eV)	168.7	168.4	167.9	169.9	169.6
A.C. (%)	1.3	0.4	0.5	2.5	2.9
N 1s B.E. (eV)	400.2	399.9	399.8	399.7	400.6
A.C. (%)	1.2	1.8	1.0	1.1	0.3
Si 2p B.E. (eV)	102.4	—	102.4	103.5	103.7
A.C. (%)	1.1	NSP ^d	1.3	2.3	17.4
F 1s B.E. (eV)			688.9	—	—
A.C. (%)			4.1	NSP	NSP
Ca 2p B.E. (eV)			347.6/351.1		348.5/351.0
A.C. (%)			1.3		1.0
Al 2p B.E. (eV)			74.7	75.3	—
A.C. (%)			1.8	2.0	0.3

^a Binding Energy.

^b Atomic Concentration.

^c Multiple Peaks.

^d No Significant Peak.

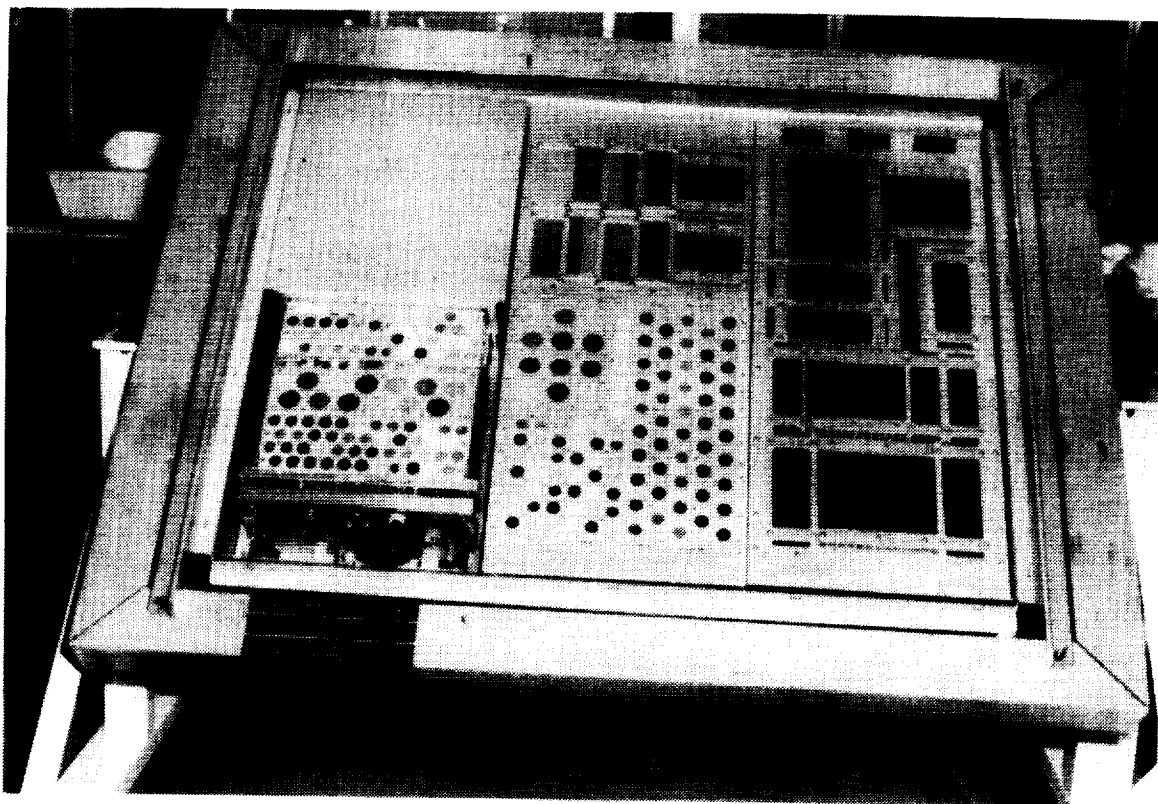


Figure 1. Preflight photograph of LDEF materials experiment tray.

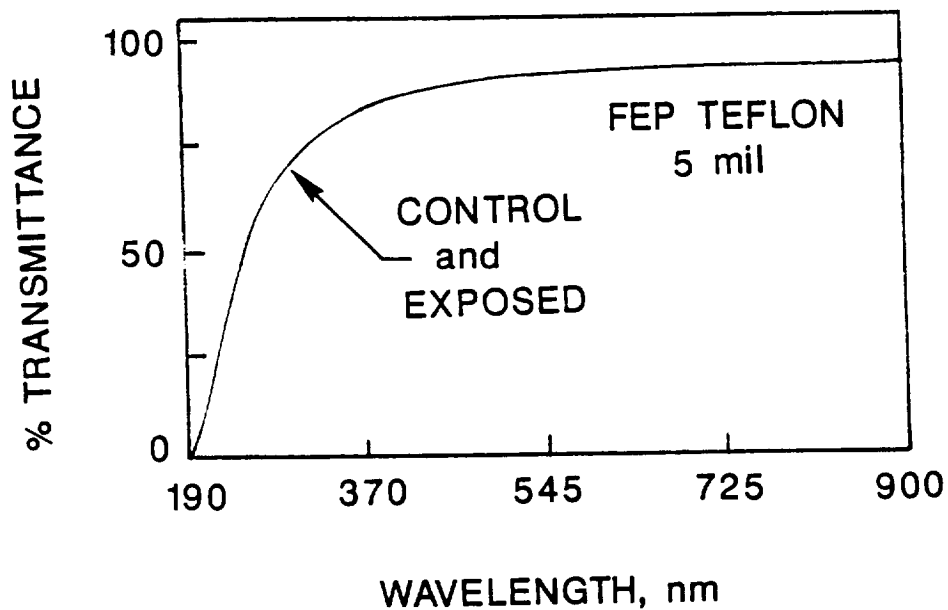


Figure 2. UV-VIS transmission spectrum of 10-month LDEF exposed FEP Teflon film.

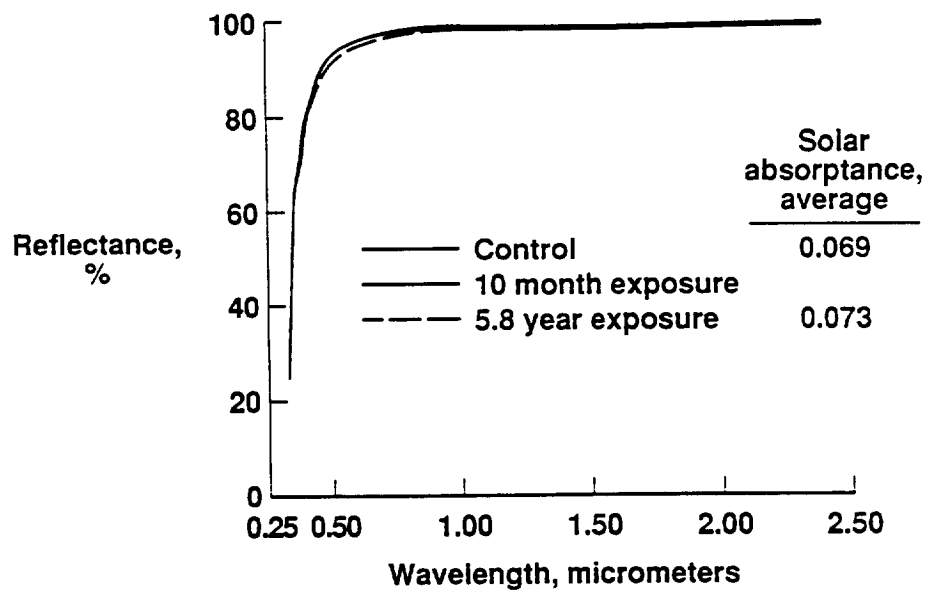


Figure 3. Total reflectance spectra for 10-month and 5.8-year exposed silvered FEP Teflon thermal control coating.

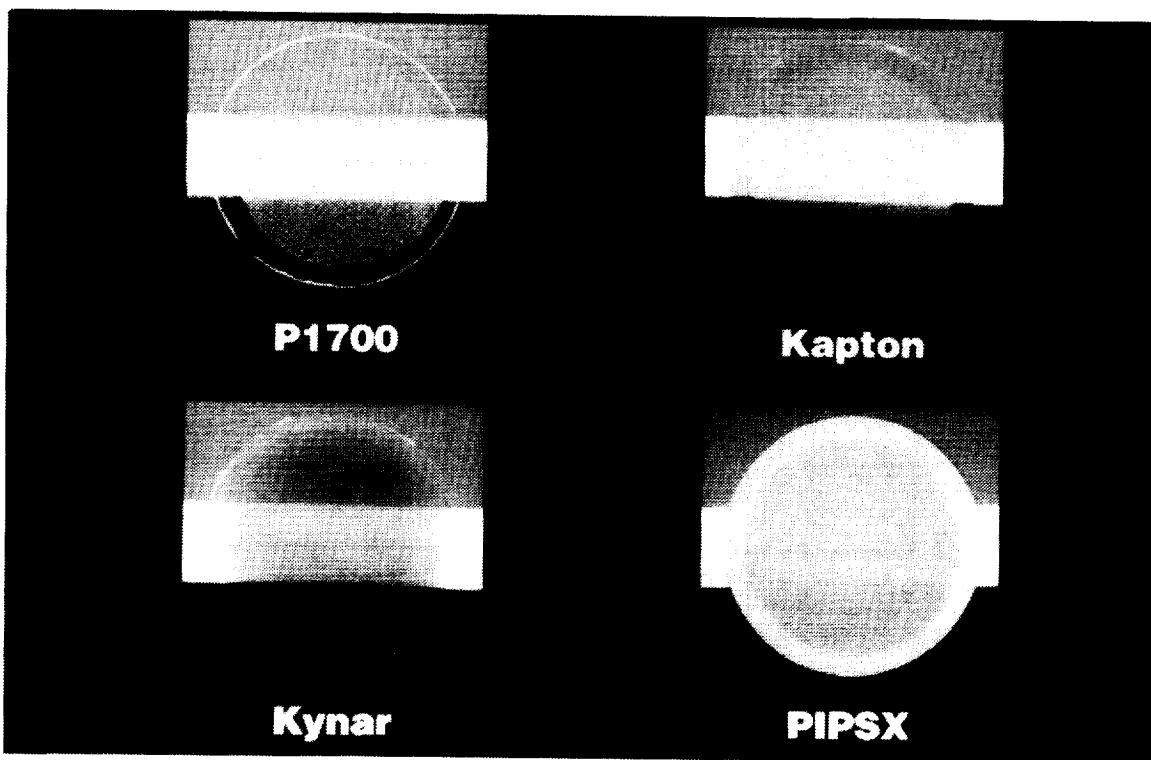


Figure 4. Photographs of 10-month exposed polymer films.

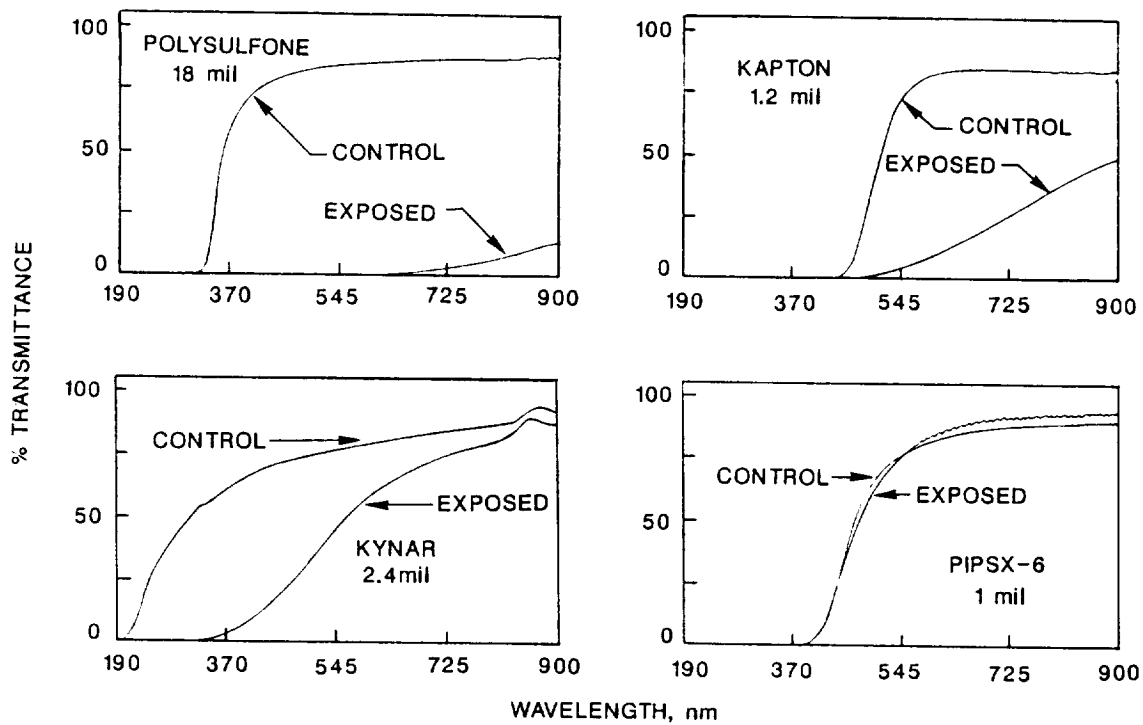


Figure 5. UV-VIS transmission spectra of 10-month exposed polymer films.

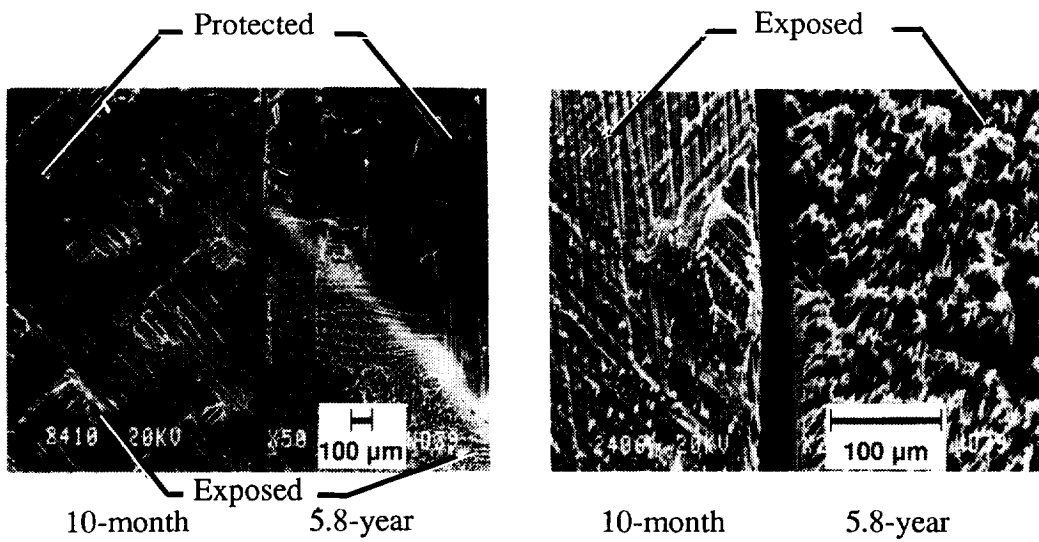


Figure 6. SEM photomicrographs of 934/T300 epoxy composites after 10-month and 5.8-year LDEF exposures.

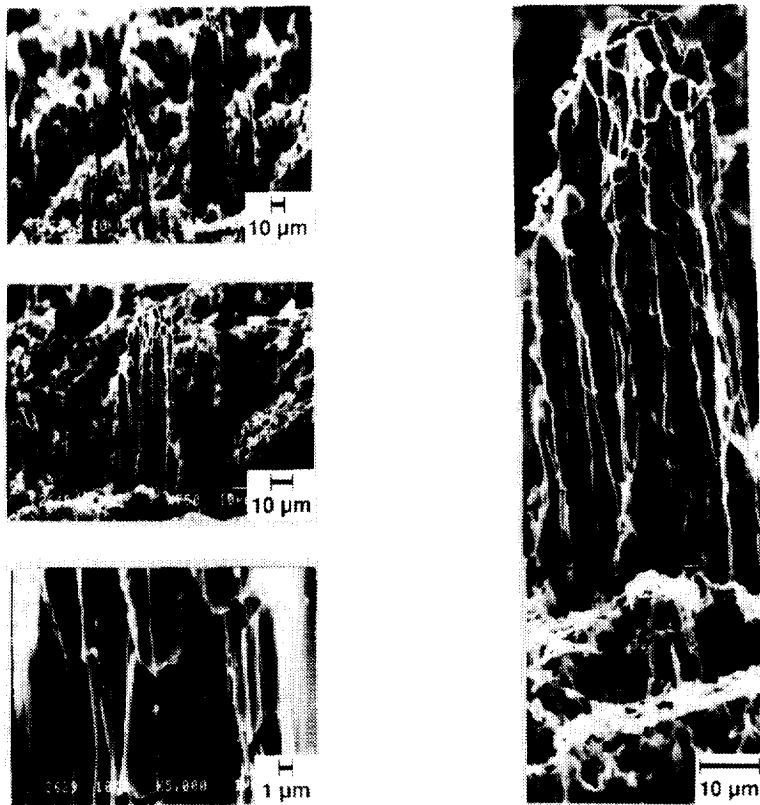


Figure 7. SEM photomicrographs of 5.8-year exposed 934/T300 epoxy composite.

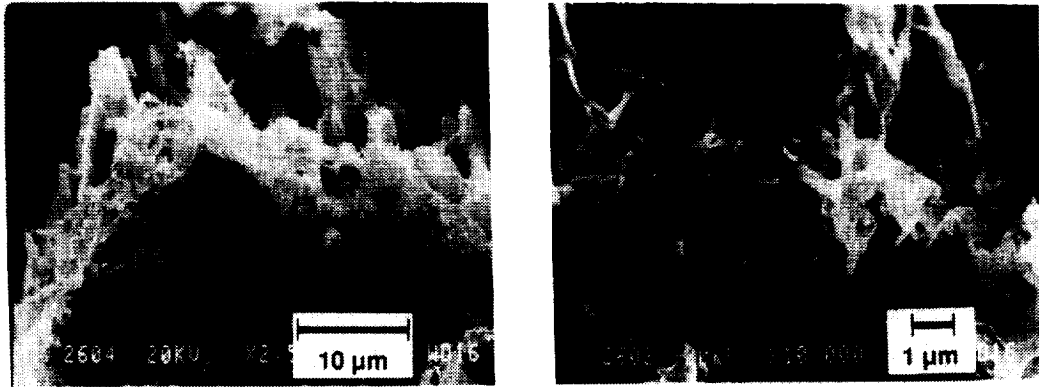


Figure 8. SEM photomicrographs of white ash or residue on exposed epoxy composite.

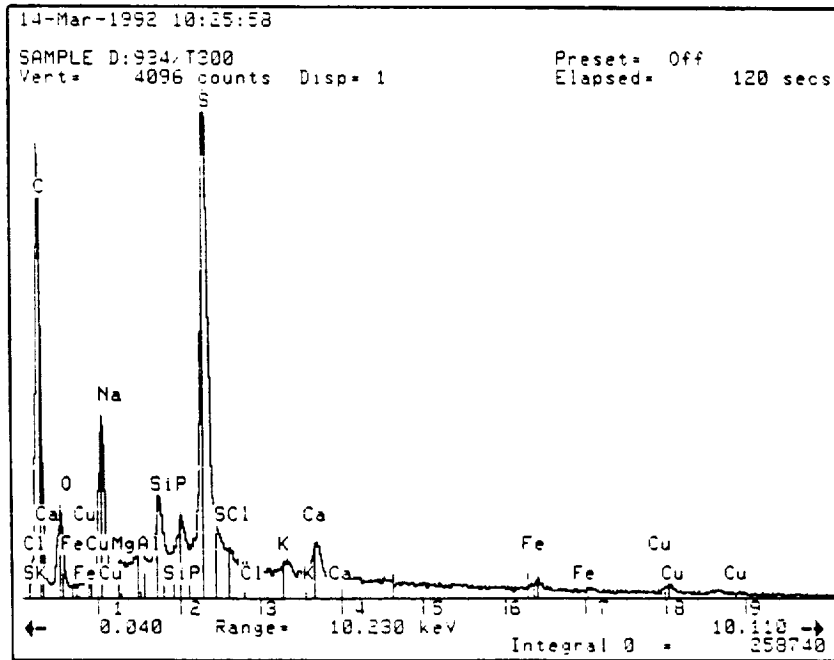


Figure 9. EDS analysis of composite residue.

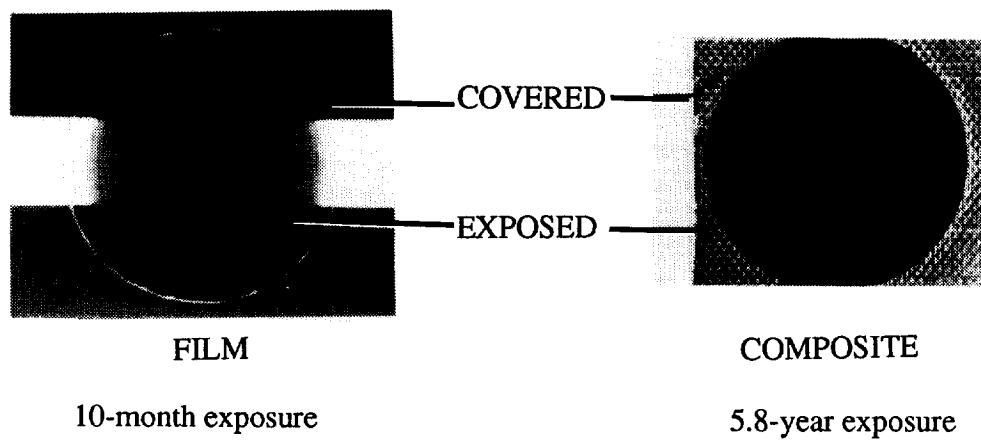


Figure 10. Photographs of LDEF exposed polysulfone film and composite specimens.

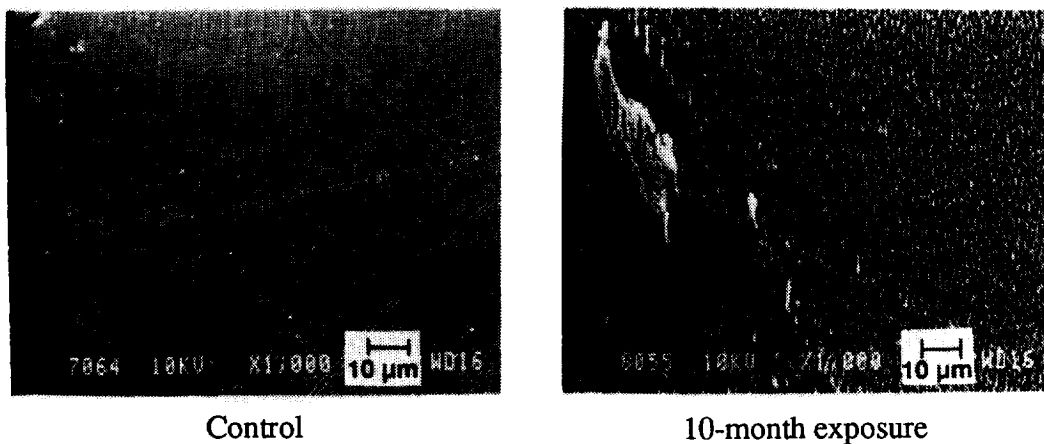


Figure 11. SEM photomicrographs of polysulfone film.

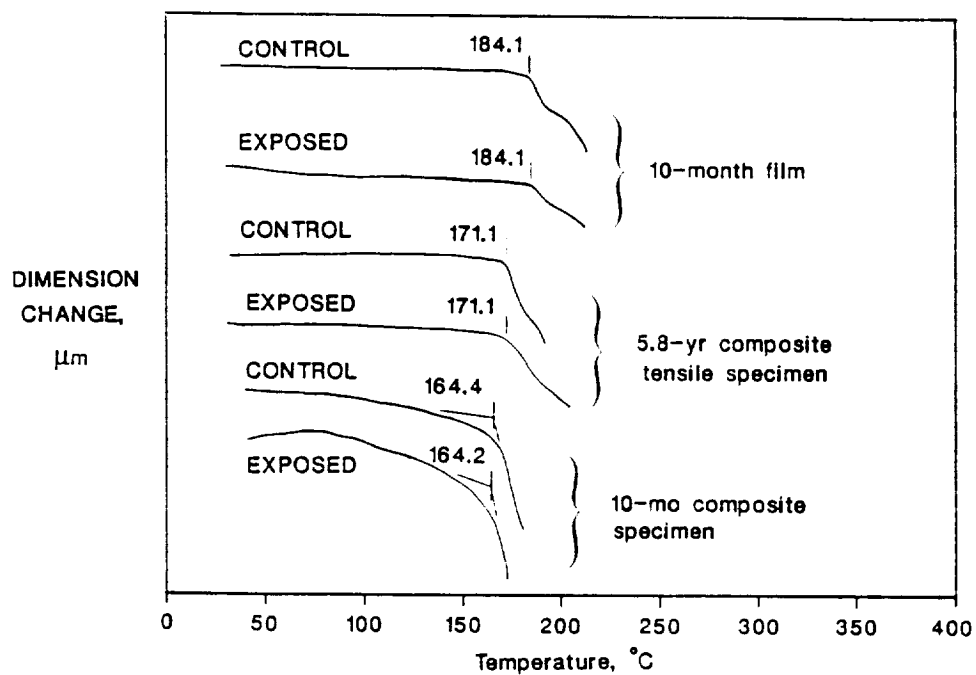


Figure 12. TMA of LDEF exposed polysulfone specimens.

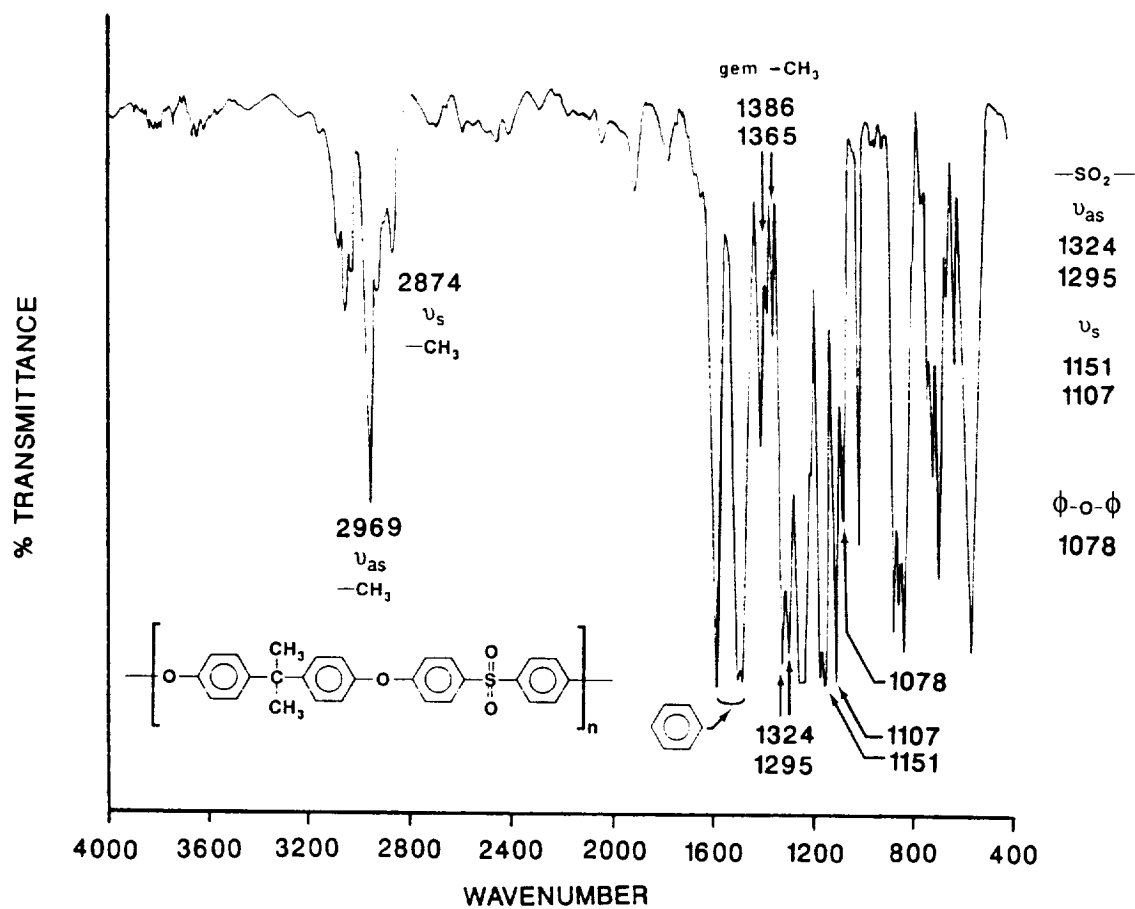


Figure 13. FTIR spectrum of a thin polysulfone film.

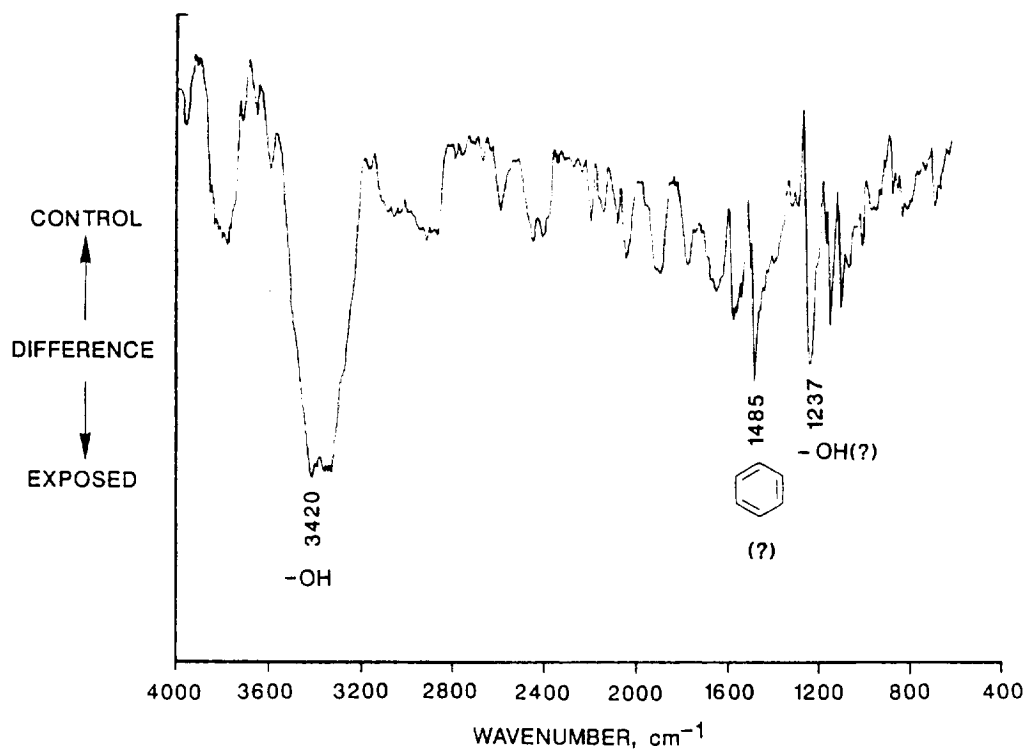


Figure 14. Subtraction DR-FTIR spectra of LDEF exposed polysulfone film specimen.

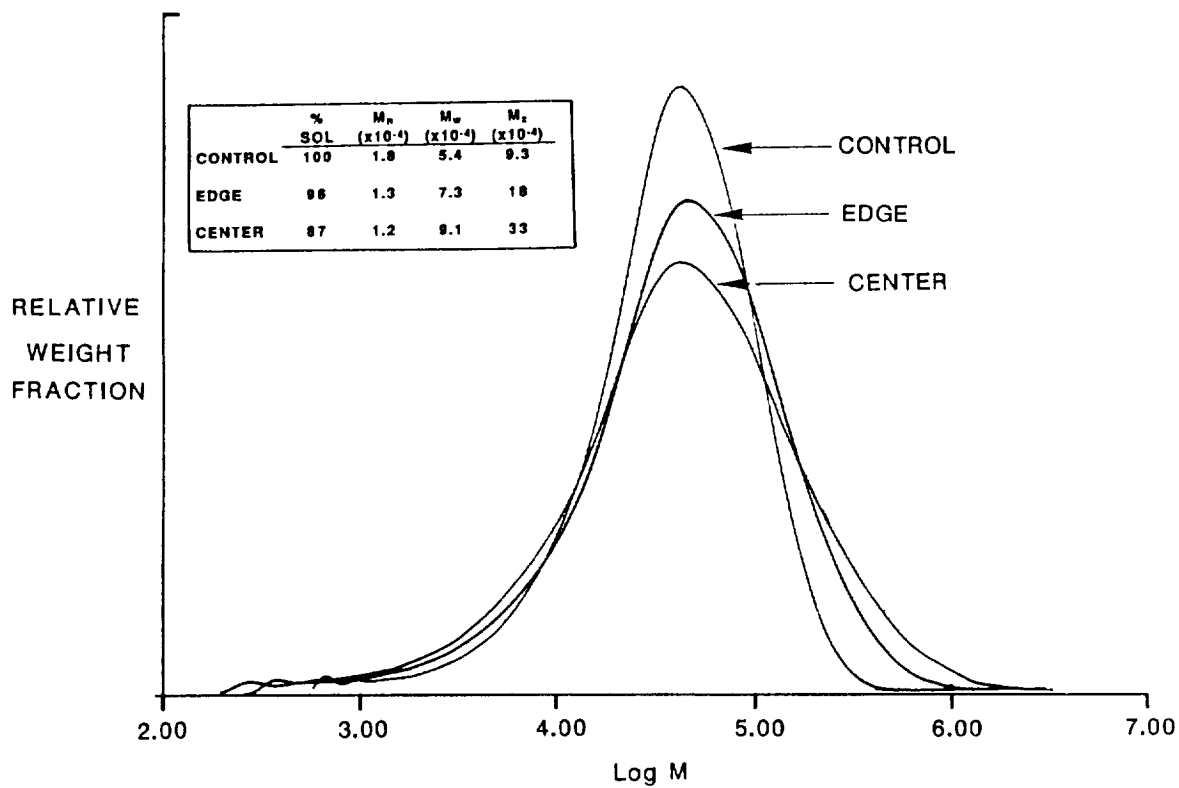
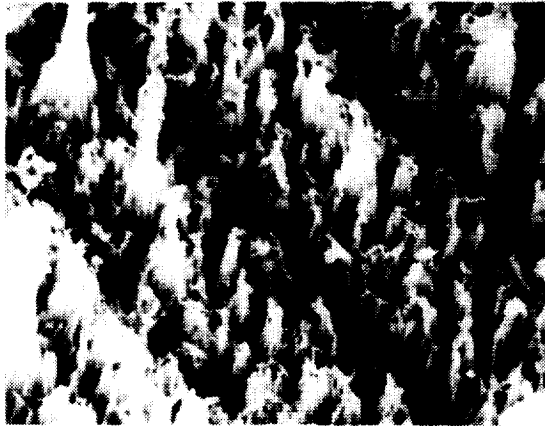
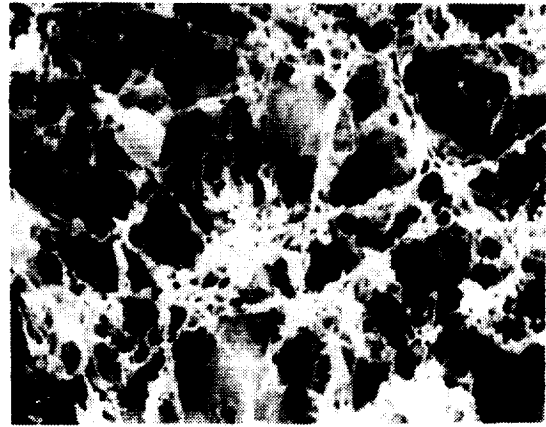


Figure 15. GPC-DV molecular weight distributions of 10-month exposed polysulfone film specimens.



a) 10 months exposure



b) 5.8 years exposure

Figure 16. SEM photomicrographs of 10-month and 5.8-year exposed polysulfone composite specimens.

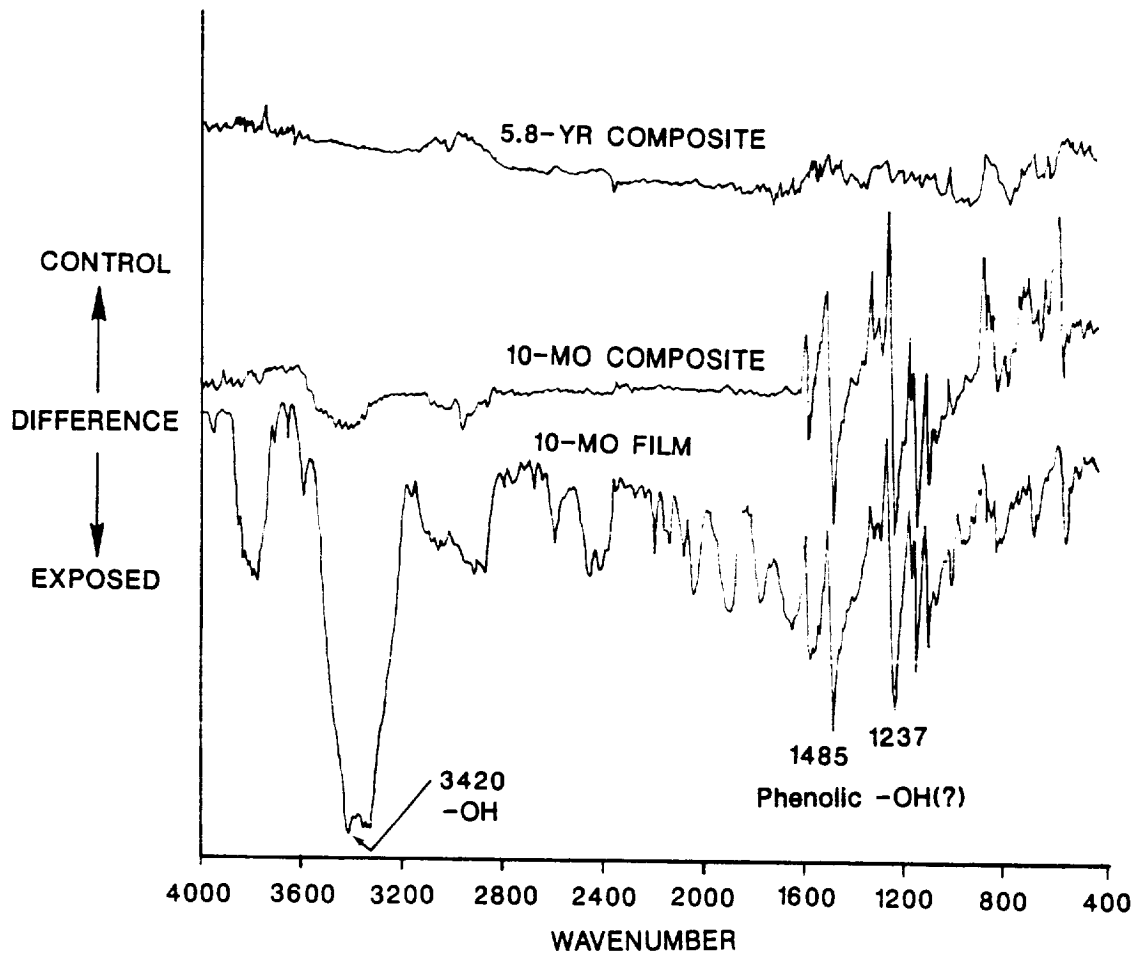


Figure 17. Subtraction DR-FTIR spectra for LDEF exposed polysulfone specimens.

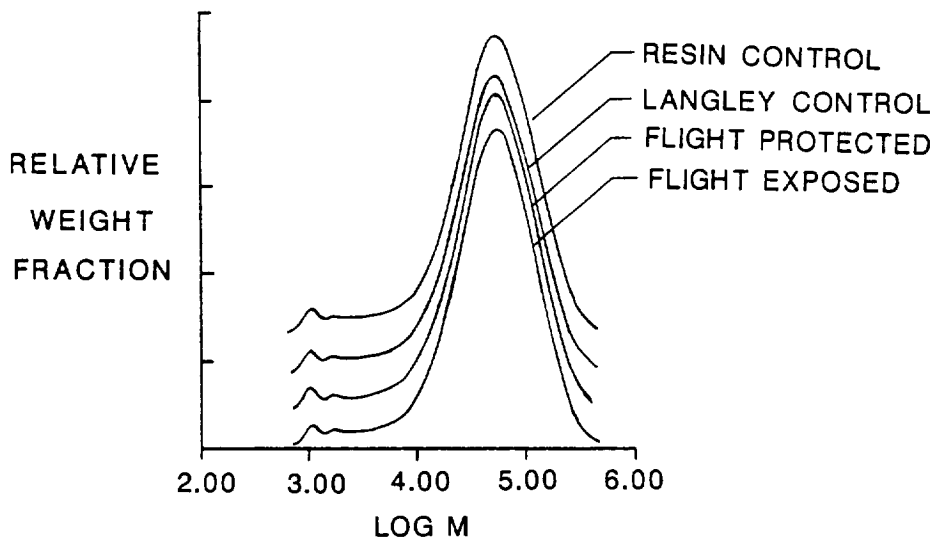


Figure 18. GPC-DV Molecular weight distributions of 5.8-year exposed polysulfone composites.

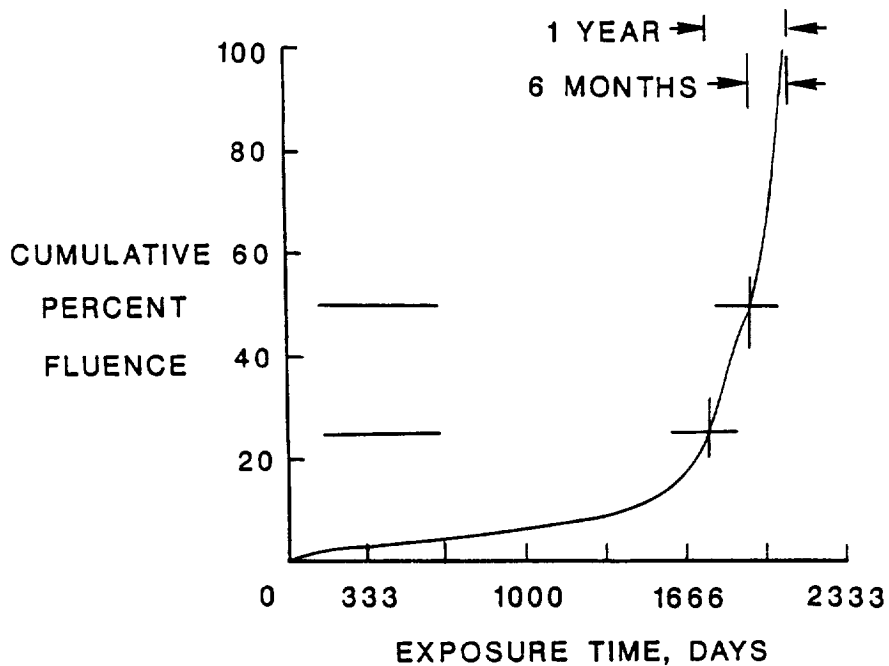


Figure 19. Approximate LDEF cumulative percent atomic oxygen fluence as a function of exposure time.

