

CHARACTERIZATION OF SELECTED LDEF - EXPOSED POLYMER FILMS AND RESINS

Philip R. Young and Wayne S. Slemp
NASA Langley Research Center
Hampton, VA 23665-5225

INTRODUCTION

The National Aeronautics and Space Administration's Long Duration Exposure Facility (LDEF) provided a unique environmental exposure of a wide variety of materials (1,2.) The effects of 5 years and 10 months of Low-Earth Orbit (LEO) exposure of these materials to atomic oxygen, ultraviolet and particulate radiation, meteoroid and debris, vacuum, contamination, and thermal cycling is providing a data base unparalleled in the history of space environment research. Working through the Environmental Effects on Materials Special Investigation Group (MSIG), a number of polymeric materials in various processed forms have been assembled from LDEF investigators for analysis at the NASA Langley Research Center. This paper reports the status of on-going chemical characterization of these materials.

CHARACTERIZATION OF SELECTED LDEF-EXPOSED POLYMER FILMS AND RESINS

The longer missions being envisioned for the U.S. Space Program are placing increased demands on materials, particularly non-metallic materials. The objective of the current work is to assess the response of selected polymeric materials to the extended LEO environment provided by LDEF. The approach has been to characterize molecular level effects in addition to more obvious visual, physical and mechanical effects. This approach should provide fundamental information for use in developing new and improved materials for long-term LEO missions.

- PROBLEM:** Inadequate knowledge of space environmental effects on materials for long duration application.
- OBJECTIVE:** Assess the response of selected polymeric materials to extended exposure to the low earth orbit.
- APPROACH:** Characterize the molecular level effects of long term exposure to space as well as visual, physical and mechanical effects.
- BENEFIT:** Fundamental information for use in developing new and improved materials for LEO missions.

OUTLINE

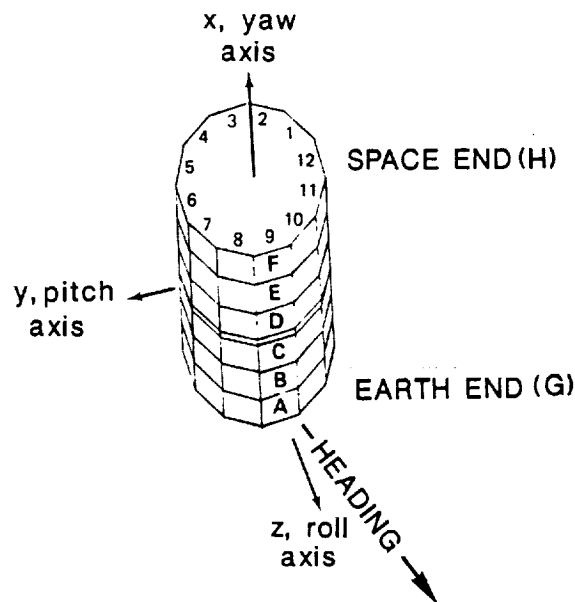
This presentation discusses several types of polymers that have either been examined, are currently being examined, or are awaiting examination. It focuses on reporting recent experimental results. For the first time, initial results from the characterization of specimens which flew inside a Row 9 Environmental Exposure Control Canister (EECC) and received only 10 months of exposure are discussed. The potential effects of the ubiquitous LDEF contamination on the performance of selected polymeric materials is presented. Finally, the possibility that some specimens may continue to change or exhibit post exposure effects is proposed.

- **Polymeric Materials**
- **Recent Experimental Results**
- **Contamination Effects on Materials**
- **Post-Exposure Effects**

LDEF SKETCH AND ORBITAL ORIENTATION

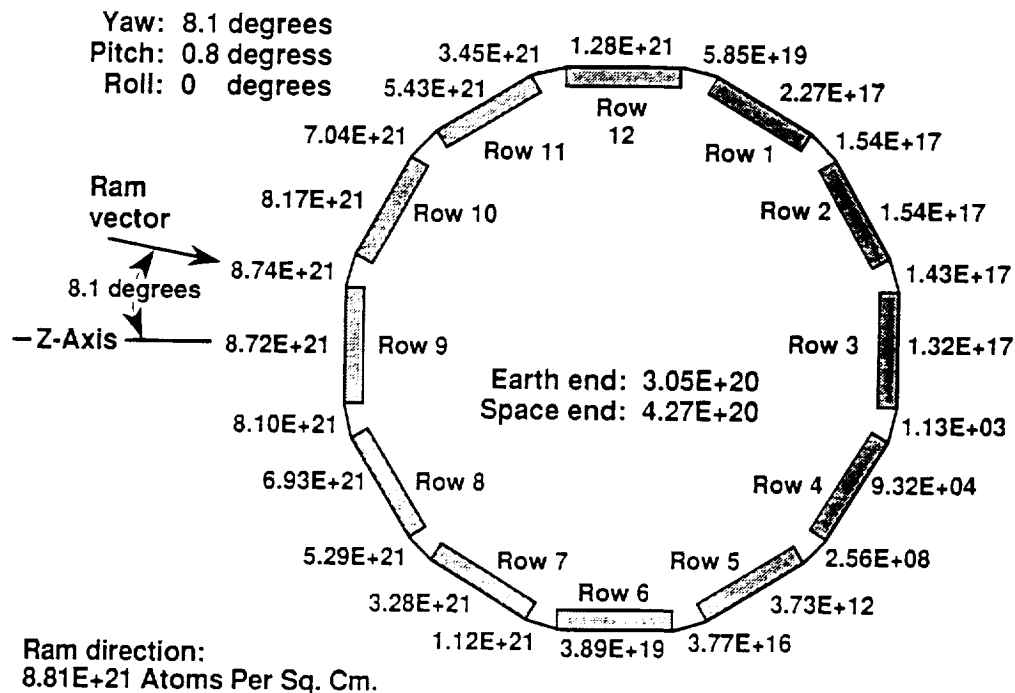
A sketch of the LDEF structure and orbital orientation is depicted in the figure. As described in Reference 1, the spacecraft was 30 feet long, 14 feet in diameter, and had 12 sides or rows with 6 experiment trays per row. One end of the gravity gradient stabilized vehicle faced space and one end faced Earth. Additional experiment trays were mounted on the space and earth ends.

Rows are numbered 1 through 12 in the figure and trays are lettered A through F. Thus, the location of specimens discussed in this report should follow from this tray and row notation scheme. For example, B9 denotes the location of specimens on Tray B at Row 9. The orbital orientation of the satellite was such that Row 9 nominally faced the RAM direction and Row 3 faced the WAKE direction. Recent LDEF supporting data analysis have determined that the actual RAM direction was 8° of yaw from the perpendicular to Row 9, in the direction of Row 10.



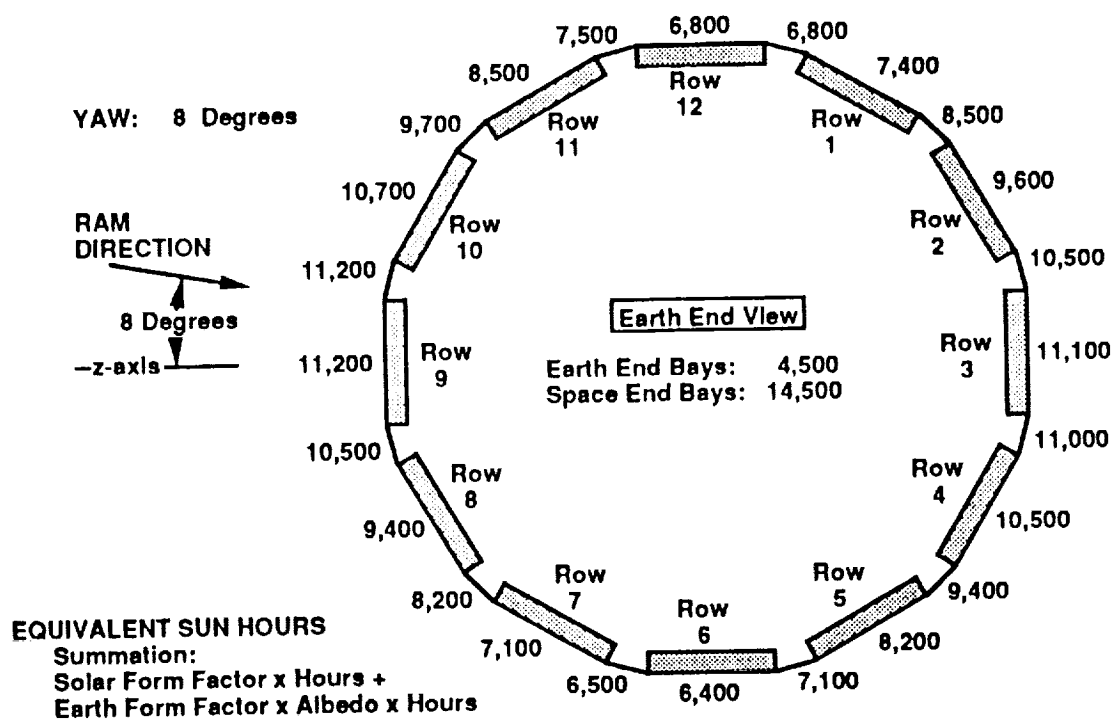
ATOMIC OXYGEN FLUENCE AT EACH LDEF TRAY

LDEF provided a very stable platform for LEO exposure of materials. The environment a specimen experienced depended on its location on the vehicle. Two significant environmental effects of concern for polymeric materials are atomic oxygen (AO) and ultraviolet (UV) radiation. The total AO fluence and equivalent UV sun hours for each LDEF row and tray have been determined by MSIG under NASA Contracts NAS1-18224 and NAS1-19247 entitled "LDEF Materials Analysis" with Boeing Defense and Space Group, Seattle, WA. The figure gives the AO fluence at end of mission for all row, longeron, and end bay locations, including the fluence received during the retrieval attitude excursion.



CUMULATIVE EQUIVALENT SUN HOURS AT END OF MISSION

Cumulative equivalent sun hours for all row, longeron, and end bay locations are given in the figure. The materials discussed in this report were exposed to additional space environmental effects. The particulate radiation (p^+ , e^- , cosmic), micrometeoroid and debris, and thermal cycling environments experienced by LDEF during approximately 34,000 orbits may be found in Reference 3. A discussion of various contamination issues may also be found in Reference 3.



POLYMERIC MATERIALS

Materials included in this study are listed in the figure. They were assembled from a number of LDEF locations. Many came from Langley's materials experiment located at B9 (4). Several LDEF Principal Investigators have unselfishly and generously made specimens from their experiments available to the authors.

The characterization of materials listed at the top of the figure is essentially complete. Results of this characterization have been summarized in a number of reports (5-9.) These reports contain experimental information describing how specimens were analyzed. The examination of materials at the bottom of the figure is in progress or is pending.

At first glance, only about one-fourth of this study appears to be complete. However, knowledge gained during the characterization of the initial specimens is useful as an aid in expediting the analysis of remaining specimens, thus, reducing the time required to complete that task. Where possible, future work will emphasize various solution property measurements of molecular weight and molecular weight distribution.

Characterization Complete or In-Progress

Silvered FEP Teflon
FEP Teflon
P1700 polysulfone
PMR-15
5208/934 Epoxy
Kapton
Polyimide-Polysiloxane Copolymer

Source: LDEF MSIG (various LDEF locations) and Expts. A0134/S0010
(W. Slemp)

Characterization Pending

Polystyrene
Polyvinyl toluene
Polytetrafluoroethylene
Polymethylmethacrylate
Nylon
Polyethylene terephthalate
Kapton

Source: J. Gregory, P.I.
Expt: A0114 (C9/C3)

Polyethylene terephthalate
Polyurethane
Various silicones
Kevlar
Teflon

Kapton

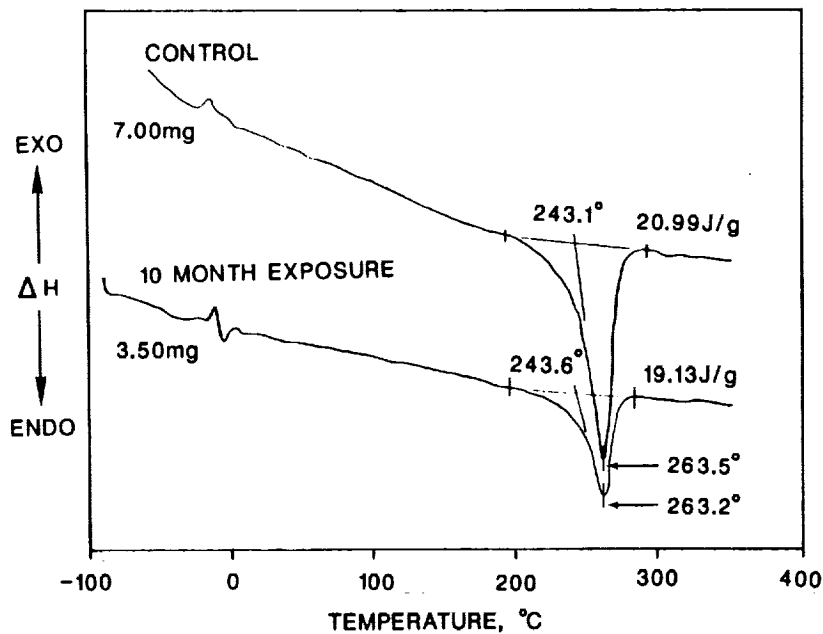
A. Whitaker, P.I.
A0171 (A8)

BTDA-ODA
6F-DDSO₂
6F-BDAF
PMDA-DAF
FEP Teflon
Kynar
Kapton

W. Slemp, P.I.
S0010 (B9)

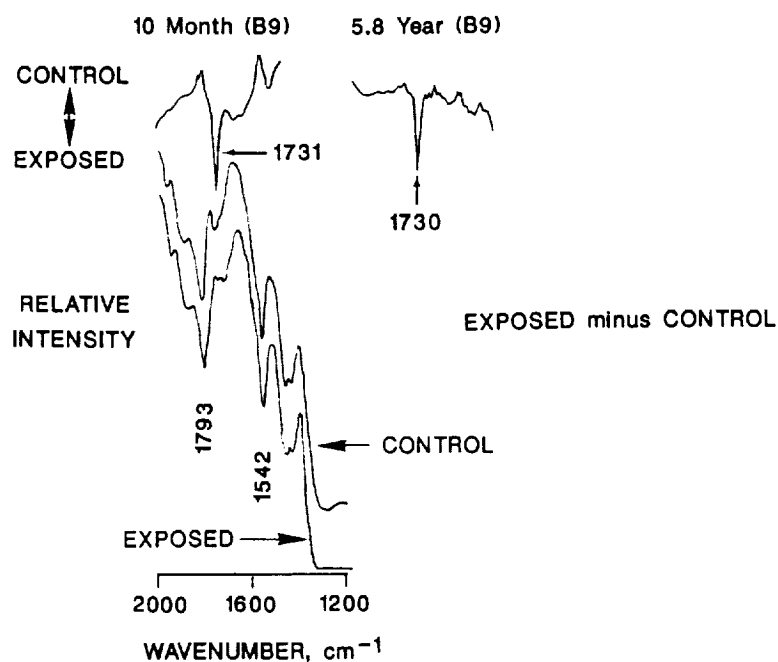
DSC THERMOGRAMS OF FEP TEFLON SPECIMENS

The 5.8-year exposure of silvered FEP Teflon thermal blankets on LDEF has been the subject of considerable research (3,8,10.) We have examined FEP specimens which received only the 10-month exposure provided by the EECC. The approximate AO fluence and equivalent sun hours experienced by these 10-month B9 specimens are 2.6×10^{20} atoms/cm² and 2,300 hours, respectively. Differential Scanning Calorimetry (DSC) thermograms of 10-month specimens are virtually superimposable over thermograms of control specimens. No significant differences were noted in low-temperature transitions, the melt point, or the heat of fusion associated with that melt. This was essentially the same conclusion derived from DSC analysis of 5.8-year FEP specimens.



DR-FTIR SPECTRA OF LDEF-EXPOSED FEP TEFLON

Standard transmission and diffuse reflectance spectra of exposed 10-month and 5.8-year FEP specimens do not exhibit interpretable differences when compared with control spectra. This suggests there are no gross differences in molecular structure of the FEP polymer backbone as a result of exposure. However, subtractive techniques reveal a weak new band around 1730 cm^{-1} in the spectrum of exposed film. This band is most likely due to the formation of carbonyl as a result of exposure to AO. It may be associated with UV-induced crosslinking of the FEP surface documented in another publication (10). The 1730 cm^{-1} carbonyl band, found in several LDEF FEP specimens, is considered to be primarily a surface phenomenon.



XPS ANALYSIS OF FEP TEFLON FILMS

X-ray photoelectron spectroscopic (XPS) analyses of two control FEP Teflon films, two 10-month specimens, and a 5.8-year specimen located at B9 are summarized below. The carbon 1s photopeaks for all samples were virtually superimposable. Multiple carbon 1s peaks associated with a crosslinked FEP surface were absent. Thus, we conclude that VUV exposure of these films was either insufficient to crosslink the surface, or that atomic oxygen had eroded the crosslinked surface away. A decrease in the CF₃:CF₂:CF ratio from 1:5:1 for control FEP to 1:4:1 after 5.8 years of exposure was noted (11).

The small amount of oxygen detected in samples after exposure probably correlates with the carbonyl discussed in the previous figure. No silicon was detected in these particular specimens. Thus, the observed oxygen was not likely associated with contamination.

PHOTOPEAK	CONTROL		10-MONTH EXPOSURE		5.8 YEAR
	SAMPLE 1	SAMPLE 2	SAMPLE 1	SAMPLE 2	
C 1s B.E. ^a (eV)	291.7	290.9	291.8	290.9	290.2
A.C. ^b (%)	32.0	31.3	32.9	30.8	30.9
F 1s B.E. (eV)	689.1	688.6	689.3	688.4	688.6
A.C. (%)	67.7	68.7	66.7	68.7	67.5
O 1s B.E. (eV)	--	--	--	532.5	532.2
A.C. (%)	NSP ^c	NSP	NSP	0.5	1.1
Si 2p B.E. (eV)		--		--	--
A.C. (%)		NSP		NSP	NSP

^a Binding Energy.

^b Atomic Concentration.

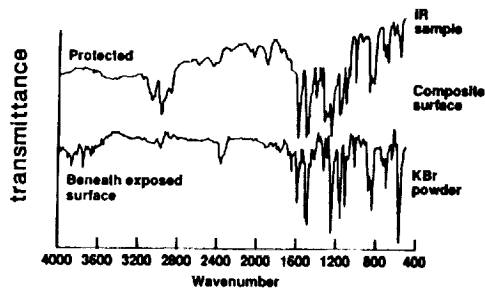
^c No Significant Peak.

LDEF - EXPOSED COMPOSITE MATERIALS

Considerable effort went into characterizing various graphite fiber reinforced polymer matrix composite materials which received 5.8 years of exposure on a Row 9 experiment (4). Since several reports have focused on these composites (5-7), only results pertinent to the present study are summarized here.

The figure gives infrared spectra, glass transition temperature, and molecular weight distribution results obtained on a series of polysulfone matrix composites. No significant differences were noted at the molecular level in these materials as a result of exposure, a general finding that also applied to epoxy matrix composites. However, the loss of about one 5 mil ply of the 4 ply composites, attributable to AO erosion, was noted. This apparently was a major contributor to the decrease in selected mechanical properties observed with exposure.

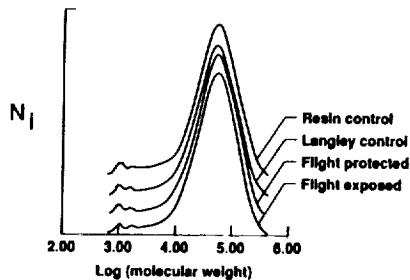
INFRARED SPECTRA



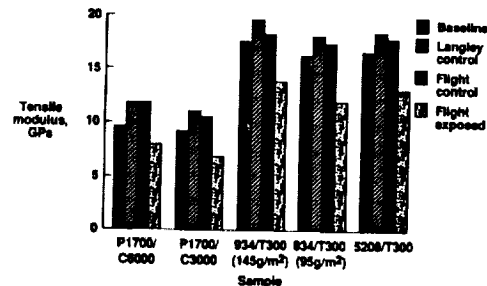
GLASS TRANSITION TEMPERATURE

Sample	T _g (°C)	Contacted Side
Langley Control	167° 167° 170° 166°	Random
Flight Protected	164° 166°	
Flight Exposed	170° 171°	Exposed side
	169° 171°	Nonexposed side

MOLECULAR WEIGHT DISTRIBUTION



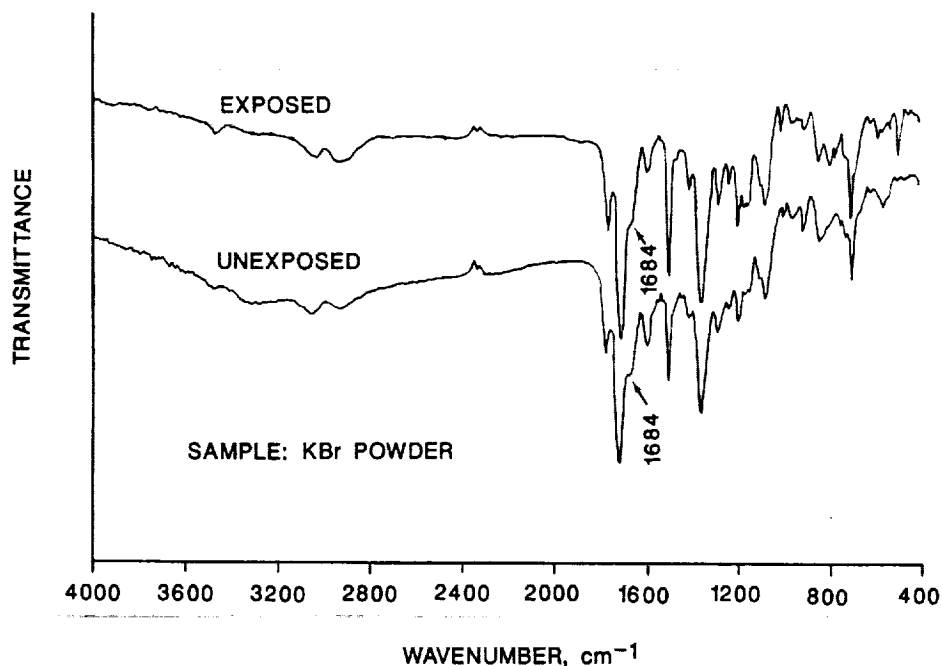
MECHANICAL PROPERTIES



DF-FTIR SPECTRA OF PMR-15/C6000 LDEF COMPOSITES

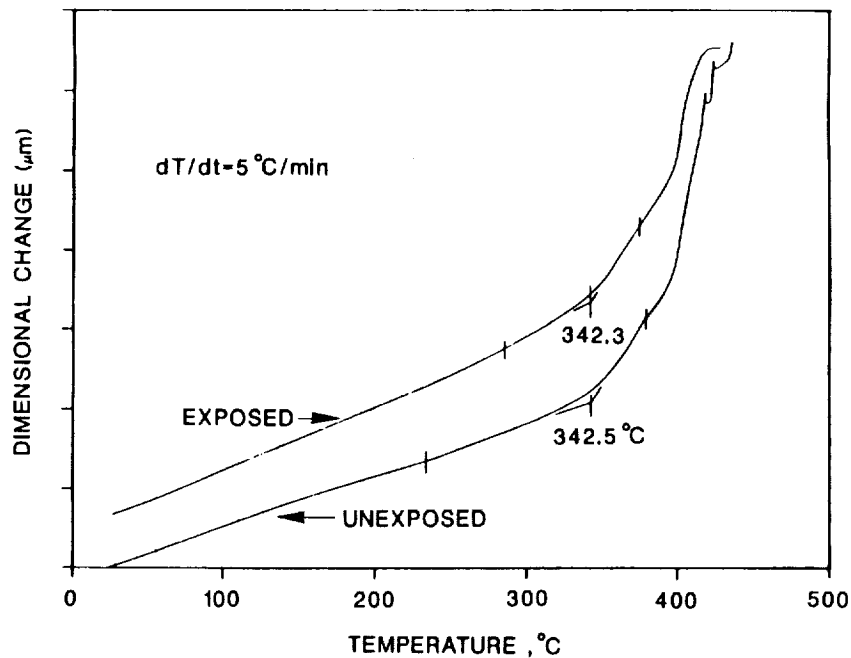
A PMR-15/C6000 composite specimen which flew on an LDEF Row 7 experiment was provided for analysis by Richard F. Vyhna, Rockwell International, Tulsa, OK. AO and UV exposure parameters for this sample can be derived from previous figures. Diffuse reflectance - FTIR spectra of exposed and protected areas of this composite are shown in the figure. The spectra are virtually identical.

A new band at 1667cm^{-1} had been anticipated in the spectrum of the exposed surface. The presence of that band would have meant that methylene groups in the amine portion of the addition end-capped polyimide resin had oxidized to carbonyl (12). The 1667cm^{-1} band is missing. The shoulder at 1684cm^{-1} is associated with the dianhydride carbonyl portion of the polymer backbone.



TMA OF PMR - 15/C6000 LDEF COMPOSITES

The PMR - 15/C6000 composite specimen from Row 7 was also examined by Thermal Mechanical Analysis (TMA) in the expansion mode. For the analysis, a probe is placed in contact with the composite surface. The temperature of the specimen is then increased, and any displacement in the probe is carefully noted. The inflection at 342°C in the curves for unexposed and exposed samples is indicative of the glass transition temperature (T_g) of the matrix resin. Since 342°C is an acceptable T_g for properly cured PMR - 15 (13), we conclude that the T_g of this material was not affected by 5.8 years of exposure. However, AO induced resin loss was noted with this specimen.

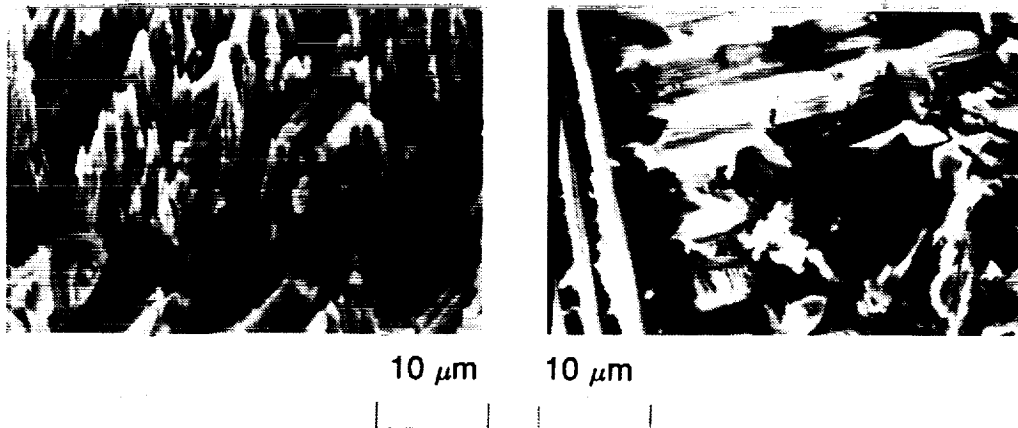


C-5

SEM OF LDEF - EXPOSED 5208/T300 COMPOSITE

As previously discussed, we have found no significant differences in matrix resin chemistry in composites which survived 5.8 years of exposure. Any molecular level changes resulting from exposure must have been lost in the layer of materials eroded away by atomic oxygen. The scanning electron microscope (SEM) analysis of a Row 9 5208/T300 epoxy specimen is shown in the figure. The loss of both resin and fiber are apparent in the figure, as are cracks in fibers. Resin/fiber content measurements show greater than 10% resin loss in Row 9 composites. Microscopic analysis shows that the top ply of 4-ply specimens has been severely eroded. The SEM in this figure is typical of the behavior observed for other examined Row 9 composite specimens.

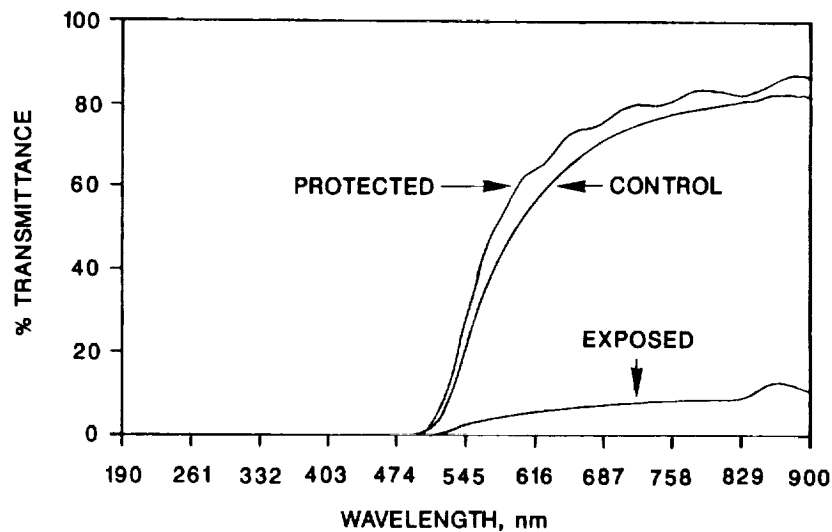
$[\pm 45]_s$



UV - VIS SPECTRA OF LDEF - EXPOSED KAPTON FILM

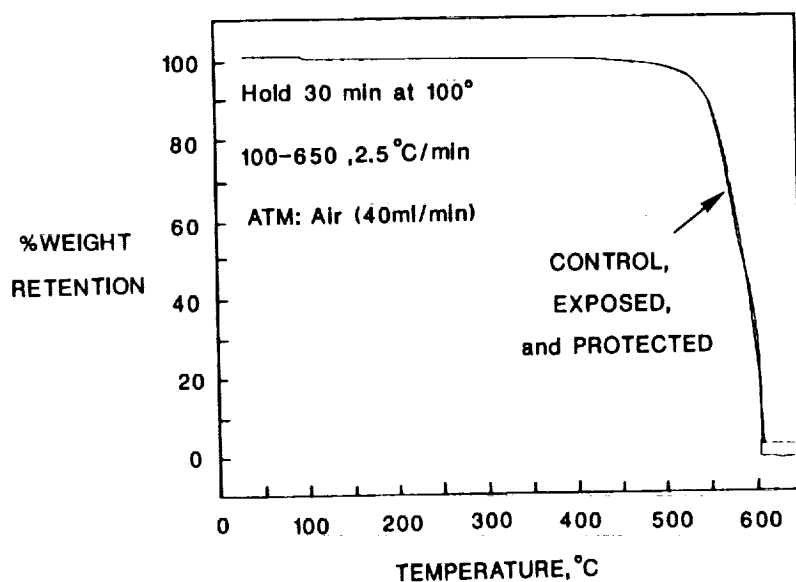
Several 5-mil Kapton film specimens which flew on the space end (H7) of LDEF and received 5.8 years of exposure were provided by James B. Whiteside, Grumman Aerospace Corporation, Bethpage, N.Y. AO and UV exposure for this location can be derived from previous figures. Due to their orientation in the experiment tray, AO exposure was perpendicular to the edge of the film and parallel to its surface. These specimens were of particular interest because of their unique AO and VUV exposure. They have been extensively studied by a variety of characterization techniques.

The figure shows UV-VIS transmission spectra of exposed film, film which flew protected from direct exposure, and a control film. No explanation is offered for slightly less transmission observed for the control specimen compared to the protected specimen. The significant decrease noted for the exposed specimen is attributed to UV degradation and AO-induced roughening of the surface. That surface exhibited a diffuse appearance.



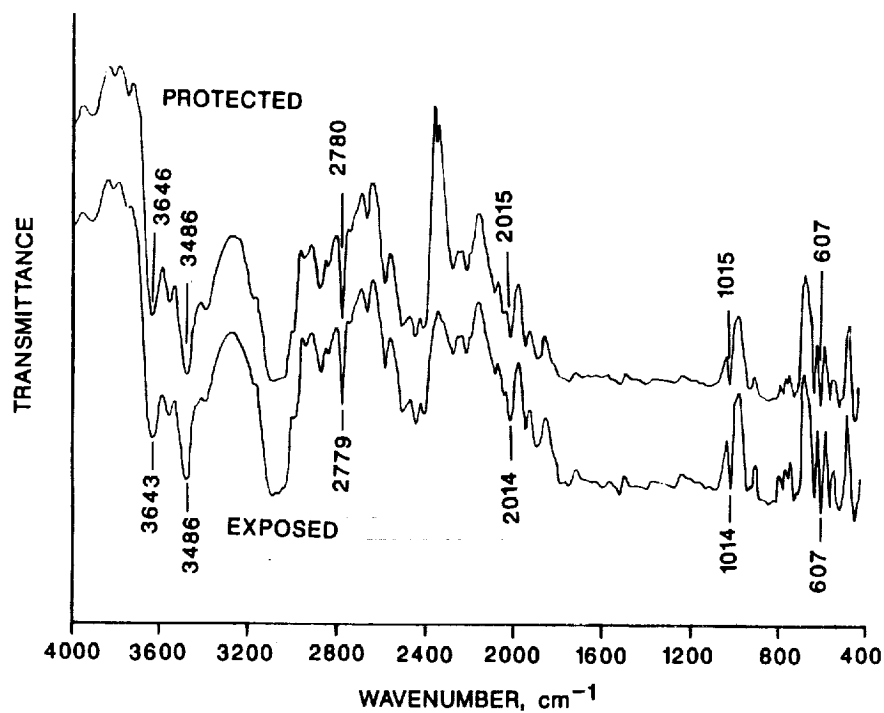
TGA OF LDEF - EXPOSED KAPTON FILM

Thermal gravimetric analysis (TGA) failed to differentiate between control, flight protected, and flight Kapton films exposed on the space end of LDEF. Weight loss curves for the three specimens were virtually superimposable. Perhaps isothermal TGA weight loss measurements, currently being performed, will detect a difference between the films.



DR - FTIR SPECTRA OF LDEF - EXPOSED KAPTON FILM

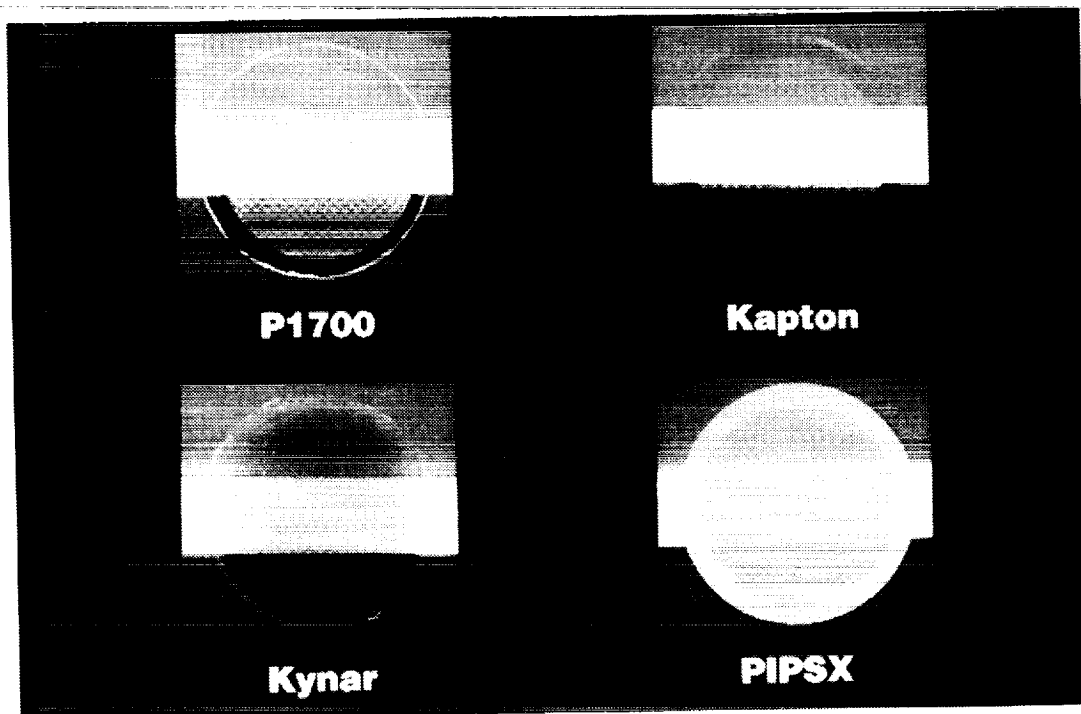
The 5-mil Kapton films from the space end of LDEF were too thick to be analyzed by standard transmission infrared techniques. Analysis by diffuse reflectance resulted in poor quality spectra. However, careful comparison of spectra in the figure reveal that no new peaks are formed as the result of exposure, no peaks are missing, and that there were no significant shifts in frequency of various bands. Thus, we conclude that the overall molecular chemistry of this polyimide has not changed as the result of exposure.



EFFECT OF 10 - MONTH LDEF EXPOSURE ON FOUR POLYMER FILMS

An analysis is in progress on several films which flew inside an EECC and received 10 months of exposure at LDEF location B9. This exposure occurred early in the LDEF flight when AO fluence was at a minimum. A photograph of four of these films is shown in the figure. Approximately 1-inch diameter films were held in place by an aluminum template with machined 0.81 inch diameter holes.

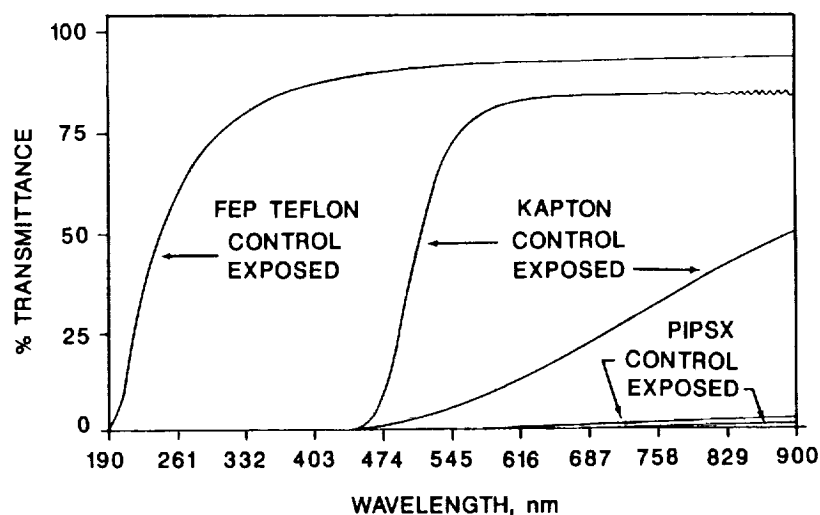
Exposed and template - protected areas are apparent in the photograph. P1700, Kapton, and Kynar are commercially available polysulfone, polyimide, and poly (vinylidene fluoride) materials. PIPSX is an experimental polyimide-polysulfone copolymer synthesized under NASA Grant NAG-1-343 with Virginia Tech.



UV-VIS SPECTRA OF 10-MONTH EXPOSED FILMS

UV-VIS spectra of three of the 10-month exposed B9 films are shown in the figure. No change was noted for the FEP Teflon film; spectra for control and exposed specimens were superimposable. The transmission of the Kapton film decreased significantly after exposure. This behavior was observed previously with Kapton from the space end of LDEF. The transmission of the opaque PIPSX film was minimal below 900 nm for both control and exposed specimens.

Research in progress on canister films is revealing molecular level effects not apparent in similar materials after 5.8 years of exposure. For example, subtractive DR-FTIR techniques show new bands in the spectrum of polysulfone film indicative of chain scission. Solution property measurements also suggest a change in selected molecular weight parameters as a result of exposure. Complete results of this research will be reported at a future date.



SEM OF POLYIMIDE - POLYSILIXANE COPOLYMER

The evidence emerging from the investigation of most polymeric materials which flew on LDEF suggests that there are no gross changes in chemistry as a result of exposure. This conclusion is based on various infrared, thermal, XPS, and solution property measurements. The subtle differences which are observed are primarily surface effects. However, substantial changes are often noted in physical and mechanical properties.

At least one material, an experimental polyimide-polysiloxane copolymer, which flew on a B9 experiment, did exhibit a significant change in chemistry after only 10 months of exposure. The figure shows SEM photomicrographs of unexposed and exposed film. A two-phase morphology is apparent. Regions that were light in appearance before exposure became dark after exposure, and dark areas became light. These two phases may have contained different amounts of the two copolymer segments which responded differently to AO exposure.

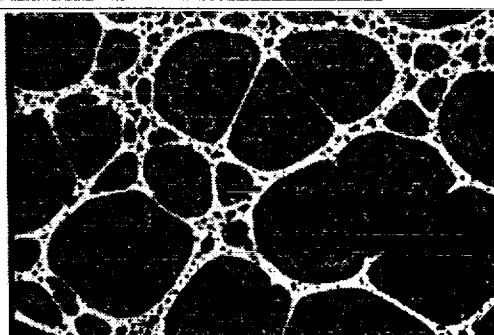
10 months of exposure

200 μm



Unexposed

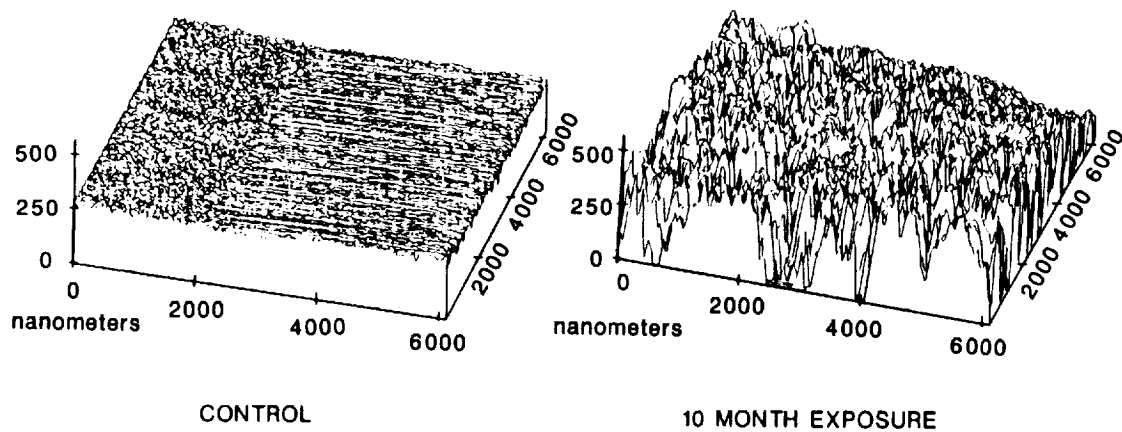
200 μm



Exposed

STM ANALYSIS OF POLYIMIDE-POLYSILOXANE COPOLYMER

Scanning tunneling microscopy (STM) analysis of control and exposed copolymer films shows considerable roughening of the surface after 10 months of flight time. Much of this roughening is assumed to be due to attack by atomic oxygen. The STM analysis of FEP teflon and Kapton film exposed for 10 months did not exhibit this type of behavior.



XPS ANALYSIS OF POLYIMIDE-POLYSILOXANE COPOLYMER

XPS analysis gave anticipated results for this copolymer. The figure summarizes data for control and exposed specimens. A 21.6% atomic concentration of silicon is noted for the control. The 102.2 electron volt binding for that sample suggests that the silicon is present as an organic silicone, as it should be. After 10 months of exposure, the surface concentration of silicon had risen to 30.8%. The 103.4eV binding energy is that of an inorganic silicate.

Atomic oxygen appears to have eroded the surface of this material to expose silicon atoms, which were then oxidized to a silica/silicate-like structure. AO has been shown to oxidize silicones to silicates (14). Further, silicates are known to be effective barriers to AO erosion (5,14,15). The behavior exhibited by this material suggests the possibility of designing AO protection into the molecular structure of selected polymers. Two additional polyimide-polysiloxane copolymers which received exposure on LDEF are currently undergoing analysis.

PHOTOPEAK	CONTROL	EXPOSED ^a
C 1s B.E. ^b (eV)	284.7	284.6
A.C. ^c (%)	54.4	16.8
O 1s B.E. (eV)	532.5	533.0
A.C. (%)	23.7	52.4
N 1s B.E. (eV)	- -	- -
A.C. (%)	NSP ^d	NSP
Si 2p B.E. (eV)	102.2	103.4
A.C. (%)	21.6	30.8

^a 10-Month Exposure.

^b Binding Energy.

^c Atomic Concentration.

^d No Significant Peak.

XPS ANALYSIS OF LDEF-EXPOSED 934/T300 COMPOSITES

Silicon has been detected as a component in the ubiquitous contamination found on LDEF (3, 5, 8). While not found on all LDEF specimens, this occurrence probably complicates the interpretation of some materials results.

The XPS analysis of two side-by-side 5.8-year exposed epoxy composites located on a B9 experiment is given in the figure. One specimen had been intentionally coated with 1000Å of nickel followed by 600Å of silicon dioxide. The other specimen was uncoated. Note that almost as much silicon was detected on the sample that was not supposed to contain silicon as was found on the sample that was supposed to contain it. The silicon on the uncoated composite undoubtedly affected the manner in which the surface was attacked by atomic oxygen.

PHOTOPEAK	600Å SiO ₂ /1000Å Ni/COMPOSITE		UNCOATED	
	COVERED	EXPOSED ^a	COVERED	EXPOSED ^a
C 1s	65.1 ^b	28.9	62.8	54.3
O 1s	29.7	47.6	24.8	33.0
N 1s	—	—	3.4	5.2
Ni 2p	4.6	11.7	—	—
Si 2p	2.4	11.8	3.4	7.5
F 1s	—	—	2.0	—
S 2p	—	—	2.0	—
Na 1s	—	—	1.7	—

^a 5.8 year exposure.

^b Atomic Concentration, %.

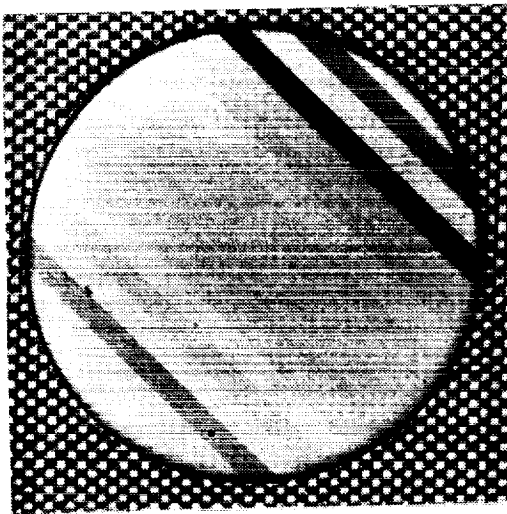
LDEF-EXPOSED 934/T300 EPOXY COMPOSITES

Photographs of the two B9 934/T300 composite specimens described in the preceding discussion are shown below. The thin SiO_2/Ni coating on the composite on the right appears to have been effective in protecting the surface from attack by atomic oxygen. The eroded area in the uncoated composite is apparent.

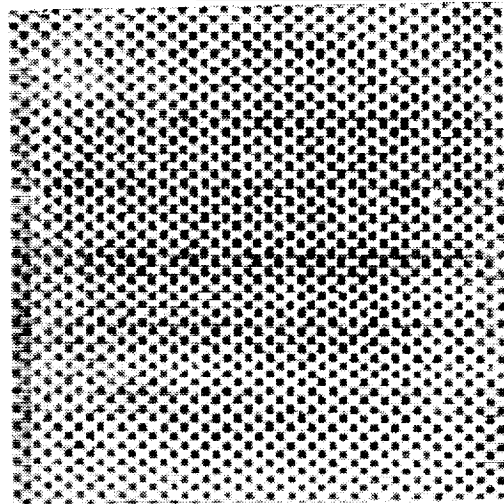
As previously noted, a small amount of silicon, presumably from contamination, was detected on the uncoated composite. This silicon probably provided some protection from atomic oxygen erosion. Thus, this specimen likely behaved differently than it would have behaved had it not been contaminated. The silicon-containing contamination no doubt caused some LDEF polymeric materials to erode less than would have been the case without contamination.

$(\pm 45)_s$, 5mil per ply

UNCOATED



600Å SiO_2 /1000Å Ni/COMPOSITE

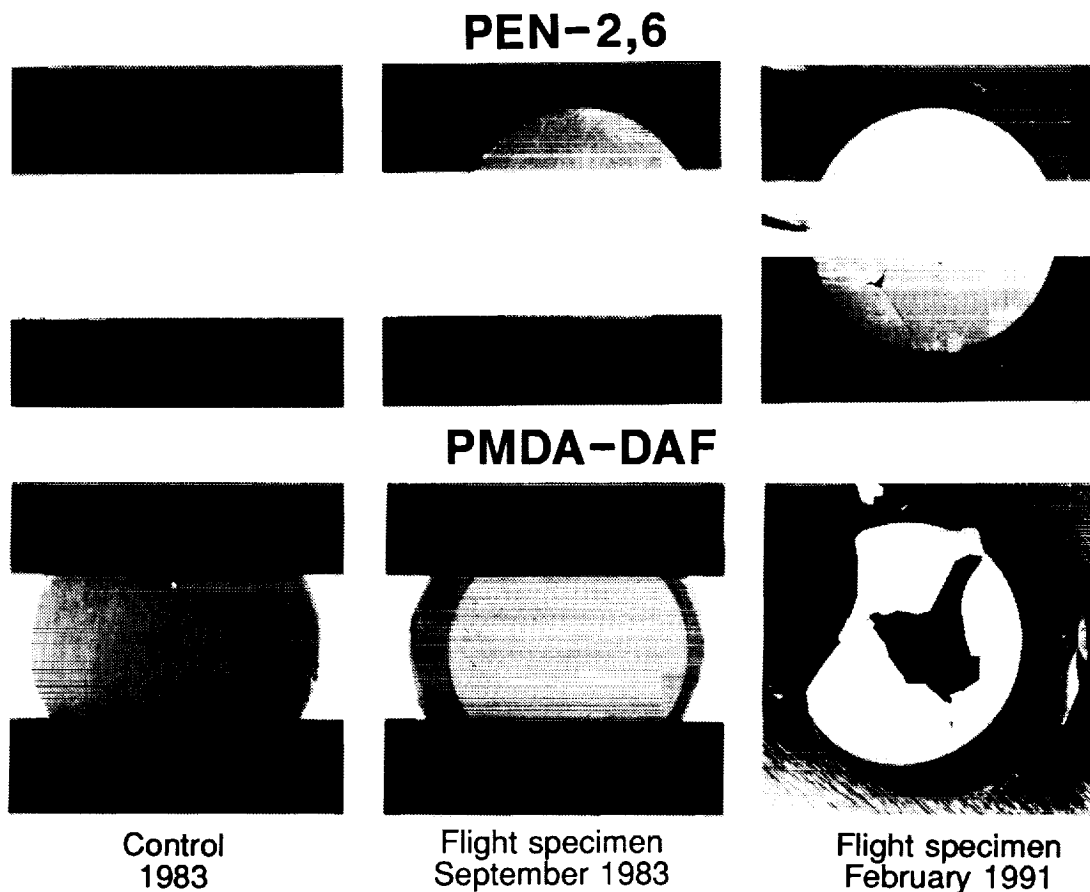


STS-8 EXPOSED POLYMER FILMS

The possibility that some polymeric materials which received exposure on LDEF may continue to degrade cannot be ignored. Environmentally exposed films and coatings have been qualitatively observed in this laboratory to change with time. An appreciation of this phenomenon may be necessary in order to analyze LDEF specimens in an efficient manner.

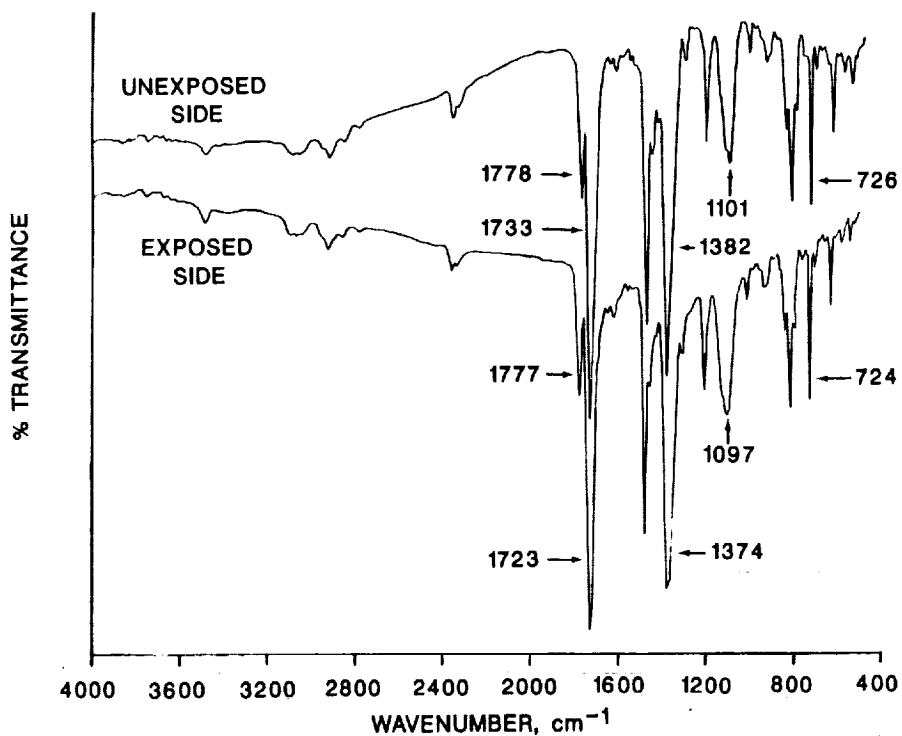
A series of thin films in a Langley experiment received 40 hours of LEO exposure in 1983 on-board STS-8 Challenger. Those films were photographed and characterized upon their return to Langley. In February 1991, the specimens were removed from a desiccator where they had been stored in tin containers. Two of four films had changed dramatically.

The figure shows photographs of control and flight specimens taken in 1983 and repeat photographs taken in 1991. PEN-2,6 shown at the top of the figure, is a state-of-the-art polyester designed to exhibit improved radiation stability (16). The film had cracked and turned opaque during storage. PMDA-DAF, shown at the bottom, is an experimental polyimide expected to exhibit unusual stability (17). That film turned opaque and lost much of its structural integrity.



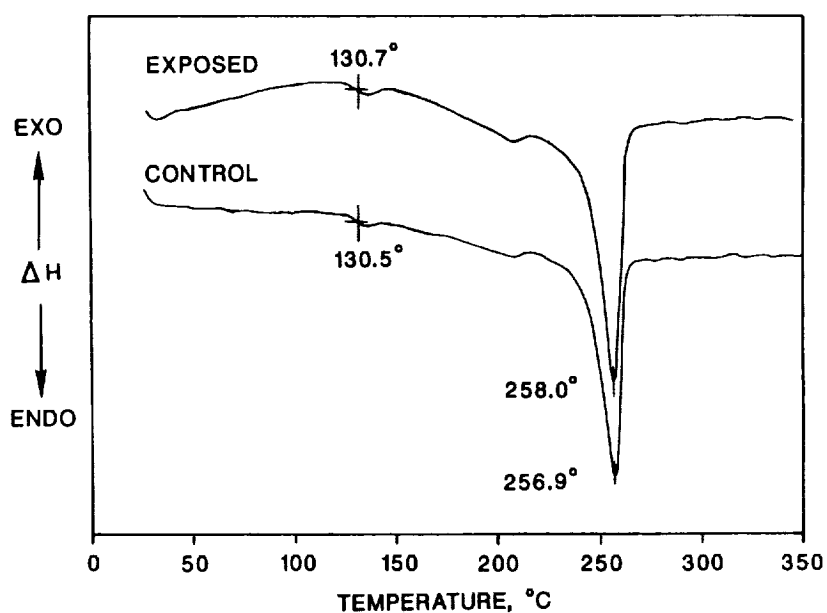
DR-FTIR SPECTRA OF STS-8 PMDA-DAF FILMS

While the physical appearance and mechanical properties of the two films had clearly deteriorated, chemical analyses to date have shown little difference between exposed and control specimens. The figure gives DR-FTIR spectra for the PMDA-DAF film. The spectra of both sides are essentially the same except for slight shifts in the absorption of five imide-related bands noted in the figure. These frequency shifts have not been interpreted but similar shifts in imide-related bands have been observed in this laboratory for polyimides that were not exposed to space.



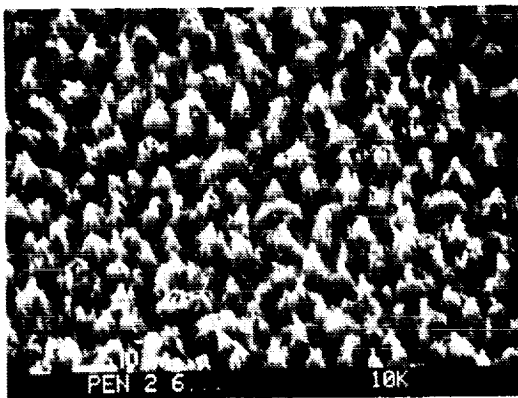
DSC THERMOGRAMS OF STS-8 PEN-2,6 FILM

DSC thermograms of exposed and control PEN-2,6 polyester film are shown below. No real differences are noted in the T_g , T_m , or the heat of fusion associated with the melt. X-ray diffraction also failed to detect a change in crystallinity in the exposed film. Thus, the increased opaqueness with age was not likely due to crystallinity effects. FTIR spectra failed to show differences in these specimens.



SEM OF STS-8 EXPOSED PEN-2,6 FILM

The figure shows the 1983 SEM analysis of the exposed polyester film, and a repeat SEM analysis conducted in November 1991. The two photomicrographs are surprisingly similar. A detailed SEM study of both the PEN-2,6 and PMDA-DAF films failed to explain the appearance of aged flight specimens. XPS analyses to date have also been inconclusive. Hopefully, pending solution property measurements on the polyester film will show differences in molecular weight and molecular weight distribution. Such a result would help explain why the films cracked. The observations made on STS-8 films keynote the urgent need to analyze non-metallic LDEF materials in an expedient manner.



1983 ANALYSIS

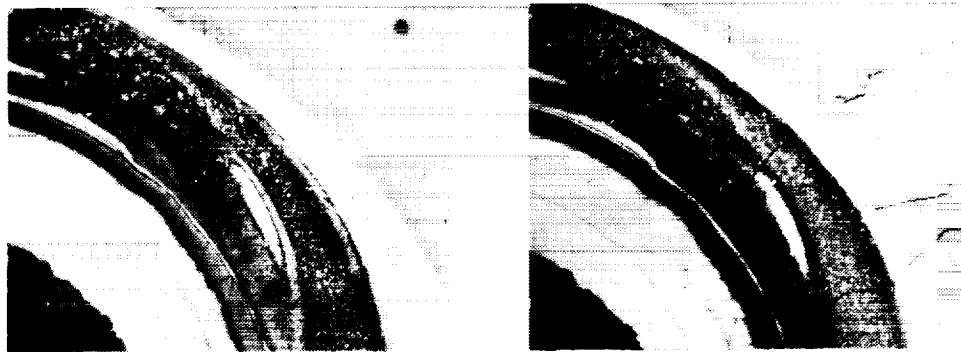


1991 ANALYSIS

MICROMETEOROID IMPACT ON Ag/FEP TEFLON THERMAL BLANKET

Selected LDEF specimens may be changing in appearance with time. A micrometeoroid impact on a Row 11 thermal blanket was photographed under magnification in April 1990, shortly after the analysis of LDEF materials began. That photograph is shown below on the left. The same area was photographed again under the same magnification in November 1991. The photograph on the right shows that cracks in the vapor deposited silver have continued to form on the silvered side of the thermal blanket material. Cracks that were present in 1990 appear to have intensified with age. The overall appearance tends to be duller.

(X100)



April 1990

November 1991

POST EXPOSURE EFFECTS FRONT COVER-LDEF THERMAL CONTROL SURFACES EXPERIMENT

J. M. Zwiener of the Marshall Space Flight Center provided two photographs taken one year apart of the front cover of the Row 9 Thermal Control Surfaces Experiment (S0069). The photographs were taken under similar lighting, angle, and distance considerations. The silvered FEP Teflon covered panel had changed in visual appearance during the year in which it was stored under prudent laboratory conditions. The diffraction patterns visible in the upper left-hand corner of the 1990 photo are real. They are not present in the 1991 photo. Brownish streaks in the coating had also intensified with age.

(S0069)



March 10, 1990

March 14, 1991

Source: J. M. Zwiener
NASA-MSFC

POSSIBLE ORIGIN OF POST-EXPOSURE EFFECTS

These distressing observations, combined with others that have been orally reported within the LDEF community, highlight the urgent need to analyze polymeric LDEF materials in an expedient manner. Otherwise, valuable information may be obscured and, in some instances, incorrect or biased interpretations may result.

Among the possible origins of these effects are residual free radicals, embrittled surfaces due to crosslinking, and unbalanced stress due to AO erosion of one film surface and not the other. Increased sensitivity to oxidation, hydrolysis, or light may be difficult to quantify. A decrease in molecular weight is considered to be a major factor contribution to the loss of structural integrity of the STS-8 films. The general area of post-exposure effects is being pursued under NASA Research Grant NAGW-2495 with the University of Queensland, Queensland, Australia.

- **Residual Free Radicals**
- **Decrease in Molecular Weight**
- **Crosslinking Embrittleness**
- **Increased Sensitivity to Oxidation and/or Hydrolysis**
- **Increased Sensitivity to Light**
- **Unbalanced Stress (Due to Surface Erosion)**

SUMMARY

The LDEF is providing a wealth of information on extended LEO exposure of selected polymeric materials. While dramatic visual effects and AO-induced resin loss have been observed, no significant change at the molecular level in many surviving polymers has been found. Due to minimum AO fluence, 10-month canister samples may exhibit surface effects not present in 5.8-year exposed samples. Potential molecular level effects with 5.8-year samples attributable to exposure have probably been eroded away by atomic oxygen .

The role that silicon in the molecular contamination can play on AO erosion rate needs to be quantified. Further, the possibility that selected specimens may continue to degrade must be appreciated. The ultimate benefit of continued LDEF-related materials research will be analytical information leading to the synthesis and development of new materials with long-term durability in the LEO environment.

LDEF is providing a wealth of information on extended LEO exposure of polymeric materials.

- **Dramatic visual effects and AO-induced resin loss.**
- **No significant change at the molecular level in many surviving polymers.**
- **Molecular contamination may bias environmental exposure results.**
- **Some specimens may continue to degrade.**

REFERENCES

1. L. G. Clark, W. H. Kinard, D. J. Carter, and J. L. Jones, ed.; The Long Duration Exposure Facility (LDEF). NASA SP-473, 1984.
2. B. A. Stein and P. R. Young, compilers; LDEF Materials Data Analysis Workshop. NASA CP-10046, 1990.
3. LDEF-69 Months in Space First Post Retrieval Symposium. NASA CP 3134 (1991).
4. W. S. Slomp, NASA SP-43, 1984, pp. 24-26.
5. P. R. Young, W. S. Slomp, W. G. Witte, and J. Y. Shen; SAMPE Intl. Symp., **36**(1), 403(1991).
6. P. R. Young and W. S. Slomp; in "LDEF-69 Months in Space First Post Retrieval Symposium," NASA-CP 3134, XXXX (1991).
7. W. S. Slomp, P. R. Young, W. G. Witte, and J. Y. Shen; Ibid.
8. P. R. Young and W. S. Slomp; NASA TM 104096, December 1991.
9. P. R. Young, W. S. Slomp, and C. R. Gautreaux; SAMPE Intl. Symp., **37**, 159, (1992).
10. A. E. Stiegman, D. E. Brinza, M. S. Anderson, T. K. Minton, E. G. Lave, and R. H. Liang; JPL Publication 91-10, May 1991.
11. NASA Grant NAG-1-1186, Interim Report V, Virginia Polytechnic Institute and State University, Blacksburg, VA.
12. P. R. Young and A. C. Chang; SAMPE Intl. Symp., **23**, 538(1988).
13. R. H. Pater; SAMPE Journal, Nov./Dec., 17 (1981).
14. M. J. Meshishnek, W. K. Stuckey, J. S. Evangelides, L. A. Feldman, R. V. Peterson, G. S. Arnold, and D. R. Peplinski; Section 5-1 to 5-33, NASA TM 100459, Vol. II, 1988.
15. W. S. Slomp, B. Santos-Mason, G. F. Sykes, Jr., and W. G. Witte; Section 5-1 to 5-15, NASA TM 100459, Vol. I, 1988.
16. V. L. Bell and G. F. Pezdirtz; J. Sci. Polym. Chem. Ed., **21**, 3083(1983).
17. V. L. Bell; J. Polym. Sci. Polym. Chem. Ed., **14**, 225(1976).

