(NASA-CR-192572) PROPERTY CHANGES INDUCED BY THE SPACE ENVIRONMENT IN COMPOSITE MATERIALS ON LDEF: SOLAR ARRAY MATERIALS PASSIVE LDEF EXPERIMENT A0171 (SAMPLE) (Camber Corp.) 18 p

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Property Changes Induced By The Space Environment In Composite Materials On LDEF: Solar Array Materials Passive LDEF Experiment A0171 (SAMPLE)

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

FOREWORD

This report has been prepared for reissue with minimum editing as a NASA TM. Camber Corporation wishes to acknowledge the help of all NASA personnel who cooperated in its preparation, especially Dr. Ann Whitaker, Ms. Miria Finckenor, and Ms. Frieda Summers.

SUMMARY

Surface modifications to composite materials induced by long term exposure in low earth orbit (LEO) were dominated by atomic oxygen erosion and micrometeoroid and space debris impacts. As expected, calculated erosion rates were peculiar to material type and within the predicted order of magnitude. Generally, about one ply of the carbon fiber composites was eroded during the 70 month LDEF experiment. Matrix erosion was greater than fiber erosion and was more evident for a polysulfone matrix than for epoxy matrices. Micrometeoroid and space debris impacts resulted in small (<1mm) craters and splattered contaminants on all samples. Surfaces became more diffuse and darker with small increases in emissivity and absorption.

Tensile strength decreased roughly with thickness loss, and epoxy matrices apparently became slightly embrittled, probably as a result of continued curing under UV and/or electron bombardment. However, changes in the ultimate yield stress of the carbon reinforced epoxy composites correlate neither with weave direction nor fiber type.

Unexpected developments were the discovery of new synergistic effects of the space environment in the interaction of atomic oxygen and copious amounts of contamination and in the induced luminescence of many materials.

INTRODUCTION

LDEF Experiment AO171, "Solar Array Materials LDEF Experiment" (SAMPLE), contained approximately 100 different materials and material processes with a 300 specimen complement. These underwent 5.8 years of exposure in LEO. The various elements of this experiment represent materials and components intended for high power-to-weight ratio solar arrays, although other optical and structural materials were also included. The longer than originally planned exposure of LDEF generally enhanced the experiment objective, even though organic thin films of the experiment were lost to atomic oxygen erosion. This passive experiment was configured to assess the synergistic effects of the LEO environment on mechanical, electrical and optical properties of spacecraft materials. Composite materials specifically were to be studied for mechanical property changes induced by the space environment. The severe surface modification, induced primarily by 5.8 years of exposure to atomic oxygen attack and micrometeoroid and space debris impacts, and questions associated with LDEF evolved contamination and its consequential effects, are also summarized.

Figure 1 shows the experiment after space exposure; the carbon fiber reinforced samples are mounted on Plate II, LDEF Row 8, Position A, in the lower center portion of the illustration. The glass fiber reinforced samples were mounted on Plate III, in the lower right position. As shown in Figure 2, the experiment materials were oriented at about 38° to incident atomic oxygen.

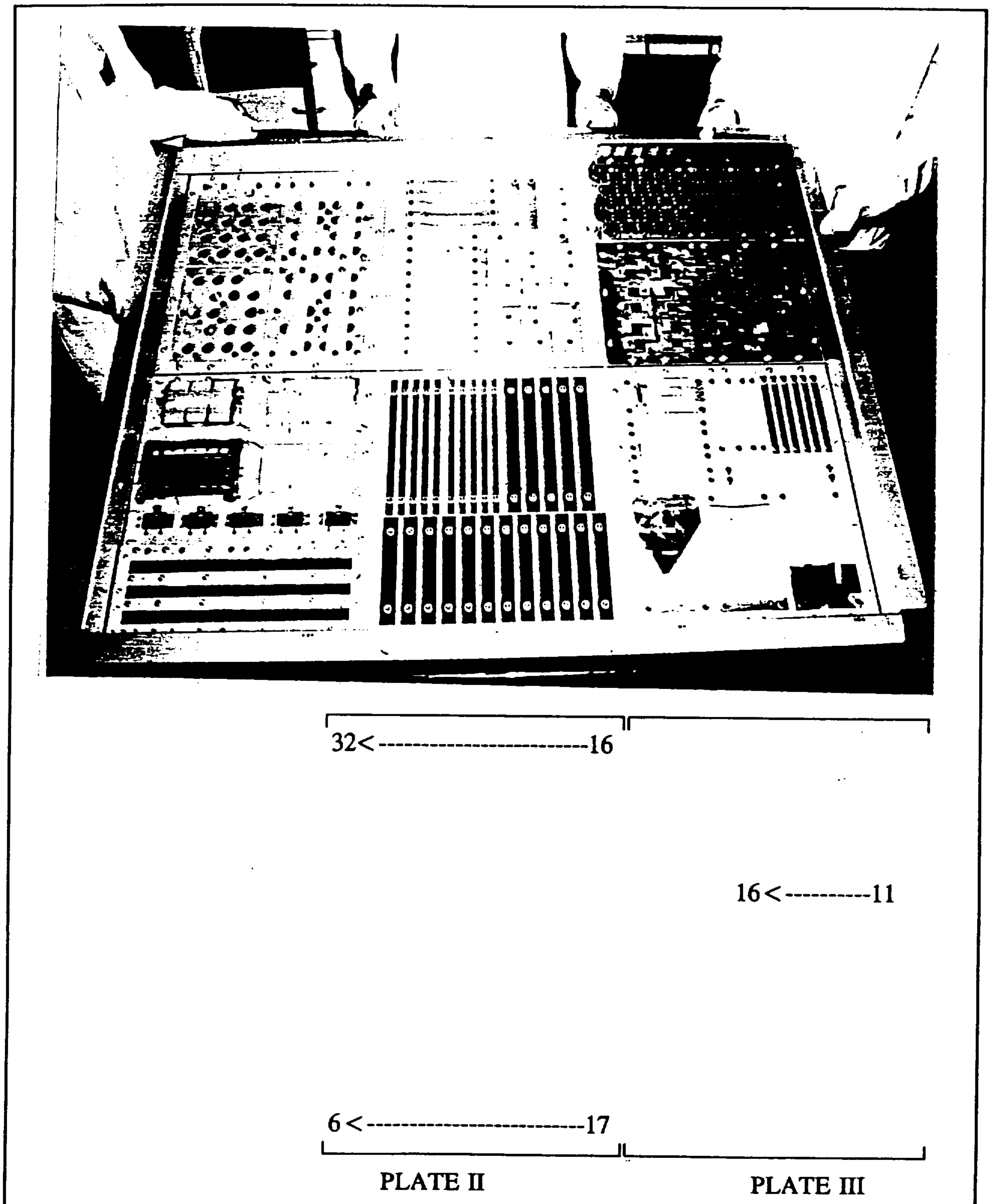


Figure 1. AO171 experiment after 5.8 years in low earth orbit. Sample location numbering is shown diagrammatically.

The total exposure to space environmental conditions is given in Table I. Temperatures reached by the composite samples can not be assessed accurately. Although well heat sunk, thermally conductive materials on Row 8 probably saw no temperature greater than 100°F, isolated materials of low thermal conductivity, mass and emittance saw wide temperature cycling. The composite samples are an intermediate case: they were thermally isolated and have low conductivity, but they maintained high emissivity and absorptance throughout the experiment. Stresses imposed on the test samples as a result of thermal cycling have not been determined but no preloads were intentionally applied.

Outgassing of materials would have occurred early in the mission, and the atomic oxygen environment was most severe during the last six months of the mission.

Table I EXPERIMENT AO171 EXPOSURE CONDITIONS

High Vacuum	10^{-6} - 10^{-7} Torr (estimated)
UV Radiation	10,471 ESH
Proton Fluence	10^{9} p^{+}/cm^{2} (0.5 - 200 MeV)
Electron Fluence	(10^{12} - 10^{18}) e^{-}/cm^{2} (0.05 - 3.0 MeV)
Atomic Oxygen	6.93 x 10^{21} atoms/cm^2
Micrometeoroids/Space Debris	2 to 5 impacts per 25 cm^2, <1mm diameter (2 to 7 impacts per composite)
Thermal Cycles	≈ 32,000 cycles (temperature unknown)

COMPOSITE TEST SPECIMENS

About one-sixth of the experiment was devoted to the exposure of composite tensile specimens. These consisted of seven groups of samples; each group contained three to six individual samples. In addition, control samples were retained in the laboratory for post-flight comparisons. All materials were vacuum baked prior to launch. Samples included strips of carbon fiber reinforced epoxy and polysulfone in three weave configurations, strips of "S" glass epoxy, and strips of "S" glass epoxy protected by aluminum tape. Figure 3 shows the carbon fiber composites held in place on experiment plate III by bolt, nut, and Teflon washer configurations. The "S" glass epoxy group was mounted separately on plate III, but with the same configuration.

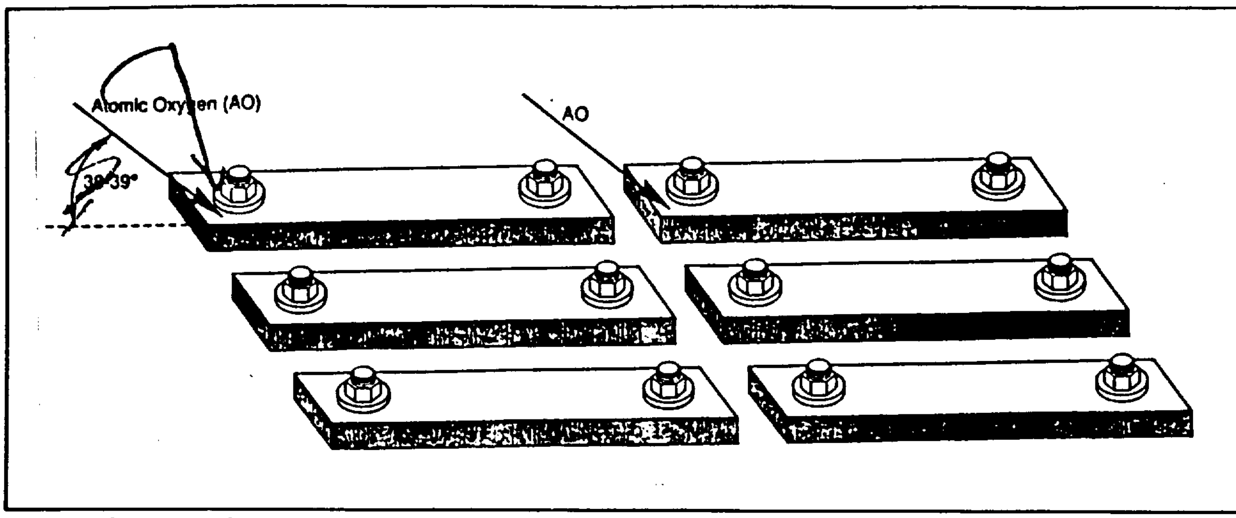


Figure 2. Atomic oxygen impingement angle on composite samples

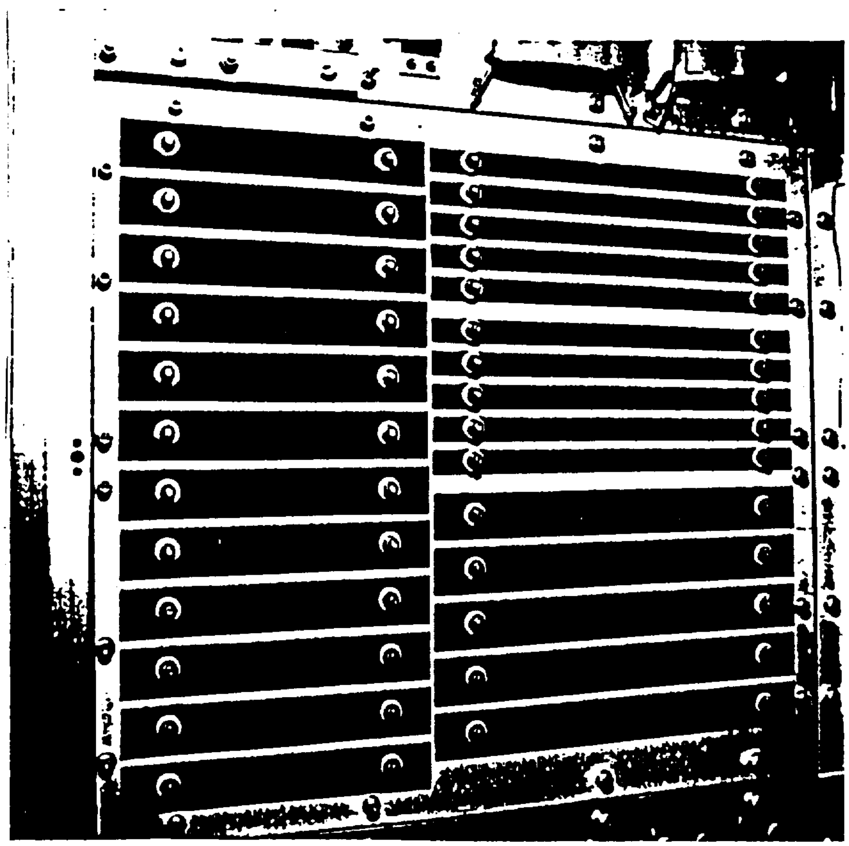


Figure 3. Carbon fiber composites mounted on experiment baseplates.

The Teflon washers served to thermally isolate the composite sample from the mounting plate and to provide an atomic oxygen protected area on the exposure surface from which surface erosion could be measured. To measure the recession of these surfaces, profilometer traces were made on each specimen in the long direction across the diameters of both protected areas. Samples also were weighed and their optical and mechanical properties measured. These measurements were compared to preflight values and to the laboratory control samples. Identification and physical characteristics of the flight specimens are given in Table II. The location of each sample is shown in the diagram of Figure 1.

General Observations

From a molecular contamination standpoint, the experiment was generally considered to be very clean with the exception of one localized area where the materials outgassed as a result of insufficient thermal vacuum bakeout prior to flight.

Particulate contamination was widespread; sources include disintegrated silicone coatings and metallized films which were transferred during the post retrieval process and splattered contaminants, probably from spacecraft fluid dumps. Dark deposits were found on the interior sides of the experiment tray at vents to the external environment consistent with the angle of atomic oxygen impingement but not consistent with sun angle. This phenomena suggests a new synergistic effect between atomic oxygen and copious contamination. The chemistry of these deposits has not been fully identified but the unbaked polyurethane paint and primer system on the interior of the LDEF surface are suspected to be the principal contributors of materials exposed to the incoming atomic oxygen at these vent locations.

A few small, scattered areas project above the recessed surface in a mesa formation and indicate that particles or other contaminants lay on the surface at the time of orbit insertion, shielding the underneath surface from atomic oxygen attack. See Figure 8.

As expected, atomic oxygen erosion and impacts dominate the surface appearance. Aluminized tape was effective in protecting the composites from atomic oxygen erosion although penetrations of the protective aluminized tape were made by small impacts. The silicone, adhesives on these tapes were well preserved and as effective as the controls. Evaluation of the tapes has been published in NASA TM-103582.

Several other phenomena were observed on this experiment that have never been reported either from flight or ground based measurements:

1. Organic materials such as polyimides, silicones, and polyurethanes were found to luminesce after flight upon exposure to far UV irradiation. Luminescence was not observed in the composite samples, but they are too dark to show luminescene photographically; their fluorescence spectra have not been analysed sufficiently to determine if their are any exposure related changes.

Table II COMPOSITE SAMPLES FLOWN ON EXPERIMENT AO171.

Group	Composite Material & References	Weave	No. of Samples	Nominal Size (") (mm)	Sample Numbers
1	HMF 322/P1700 Refs. (1,5)	±45°	5	10.5 x 0.5 x 0.058 (267 x 13 x 1.47)	Plate II 18 -22
2	HMS/934 Refs. (2,6)	0°	5	10.5 x 0.5 x 0.044 (267 x 13 x 1.18)	Plate II 28 - 32
3	HMS/934 Refs. (2,6)	90°	6	8 x 1 x 0.042 (203 x 25 x 1.07)	Plate II 6 - 11
4	P75S/934 Refs. (3,6)	90°	6	8 x 1 x 0.041 (203 x 25 x 1.04)	Plate II 12 - 17
5	P75S/934 Refs. (3,6)	0°	5	10.5 x 1 x 0.041 (267 x 25 x 1.04)	Plate II 23 - 27
6	S-901/epoxy Refs. (4,7)	0 & 90°	3	6 x 0.5 x 0.042 (152 x 13 x 1.07)	Plate III 14 - 16
7	Thermal Control Aluminized Taped S-901/epoxy Refs. (4,7,8)	0 & 90°	3	6 x 0.5 x 0.042 (152 x 13 x 1.07)	Plate III 11 - 13

References:

(1)	HMF 322 Carbon Fiber	Toray Industries, Inc., Tokyo, Japan
(2)	HMS Carbon Fiber	Courtaulds Advanced Materials, Sacramento, CA
(3)	P755 Carbon Fiber	Amoco Performance Products, Inc, Greenville, SC
(4)	S-901 Glass Fiber	Owens-Corning Fiberglass Co., Toledo, OH
(5)	Udel P-1700 Polysulfone	Union Carbide Corp, Danbury, CT
(6)	934 Epoxy	Composites Div., Fiberite Corp., Winona, WI
(7)	"epoxy" - unidentified	Air Logistics

All prepregs by Fiberite.

(8)	Aluminum tape	2 mil aluminum with 2 mils of pressure sensitive silicone adhesive SR574

2. Fibrous "ash" material was observed on carbon fiber based composite materials.

3. Silicone adhesives functioned surprisingly well after 5.8 years in space.

4. A high density of small (<1mm) micrometeoroid and space debris impacts was obvious on all materials.

5. New synergistic effects were noted where atomic oxygen and copious amount of contamination were interactive.

Surface Effects

Optical Effects. The most obvious visible effect noted on these composites is blackening and increased surface diffuseness, which is commonly observed in materials exposed to orbital atomic oxygen; see Figure 4 and Figure 5. Emissivity and absorptance increased modestly for all unprotected samples, by 12% and 6.6%, respectively, for the carbon fiber reinforced specimens. Data for the glass reinforced samples is too sparse to support a statistically valid numerical evalution of the changes. Table III summarizes optical measurements.

Mass Loss. All composites including those covered by aluminized thermal control tape lost weight. This mass loss is attributed to atomic oxygen attack except for the small mass loss of the aluminized tape covered composites, which may have lost adhesive at the tape edge due to long term thermal vacuum exposure. All of these composites were thermal vacuum baked at 100°C and 10^{-6} Torr for three hours prior to inclusion in the experiment, so no appreciable amount of volatiles would be expected to be removed from the fiber/matrix materials by the thermal vacuum alone. The surfaces show the type of appearance expected from atomic oxygen erosion but with two new features evident: (1) a structured thin ash aligned with the fiber layup configuration on the surface of all carbon reinforced composites, and (2) an expected erosion of matrix material far in excess of that of the fiber.

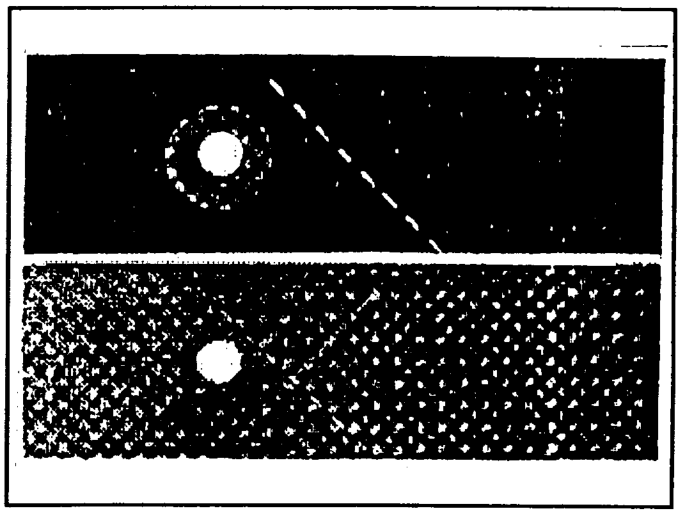


Figure 4. Comparison of carbon fiber composite specimens. Flight (top) and control (bottom).

All of the carbon fiber composites have a charred appearance and show the fiber direction as a result of preferential atomic oxygen erosion of the matrix material. The porous ash was most concentrated on the polysulfone materials.

Table III OPTICAL PROPERTIES OF AO171 COMPOSITES.

Group & Sample #s	Composite Material	Weave		Mean Solar Absorptance (limited sample)	Mean IR Emissivity (All samples)
1 Plate II 18 - 22	HMF 322/P1700	±45°	Control	0.90	0.81
			Flight	0.94	0.91
2 Plate II 28 - 32	HMS/934	0°	Control	0.88	0.78
			Flight	0.945	0.89
3 Plate II 6 - 11	HMS/934	90°	Control	0.87	0.78
			Flight	.95	0.90
4 Plate II 12 - 17	P75S/934	90°	Control	0.88	0.81
			Flight	0.935	0.89
5 Plate II 23 - 27	P75S/934	0°	Control	0.89	0.81
			Flight	0.94	0.89
6 Plate III 14 - 16	S-901/epoxy	0° & 90°	Control	0.72	0.89
			Flight	0.79	0.895
7 Plate III 11 - 13	Al Taped S-901/epoxy	0° & 90°	Control	0.14	0.025
			Flight	0.10	0.020

Mass loss was consistent with thickness loss with the carbon fiber based composites losing approximately 1 ply. The profilometer traces were used to calculate thickness lost. Fiber bundles are easily discernable in the traces; see Figure 6. Figure 7 shows the fractional mass loss for the carbon fiber reinforced samples carried on Plate II.

Surface erosion patterns were not inconsistent with that observed in short term exposure to LEO. Peak type structures characteristic of atomic oxygen attack and mesas protected by contamination are observable on the exposed surfaces. Matrix erosion was greater than carbon fiber erosion and most severe for the P1700 polysulfone system. Atomic oxygen reactivity

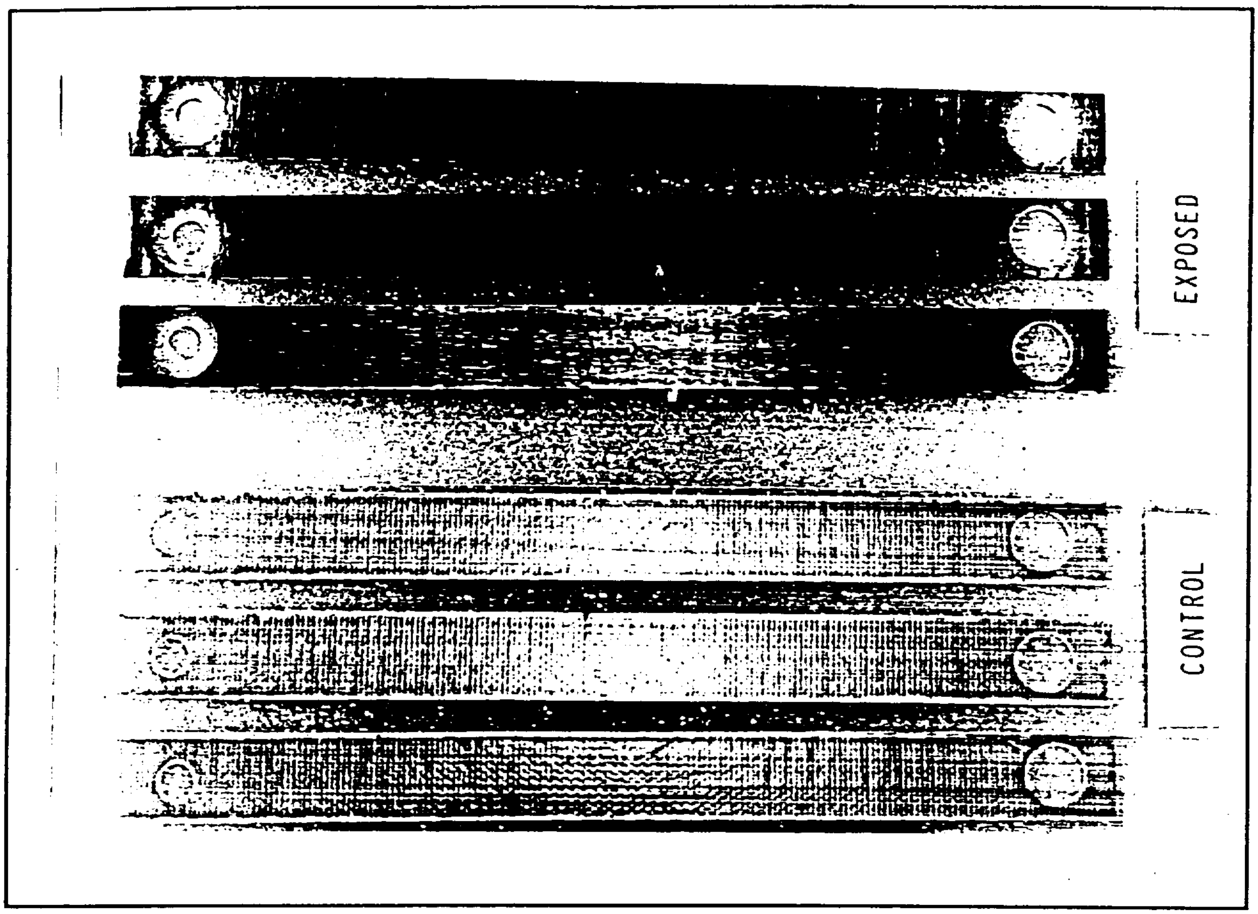


Figure 5. Comparison of glass reinforced composite samples.

numbers for carbon fiber reinforced composites were approximately twice that observed during short term shuttle exposure, and approximate short term measurements of carbon reactivity. However, short term exposure data can not be easily correlated with the long term exposure data, because the composite surfaces are rich in matrix material and, once exposed, the reinforcing carbon fibers afford some protection. Similarly, the S-glass reinforced epoxy composite become self-protected. Table IV summarizes these results.

Impact Damage. Micrometeoroid and space debris impacts were present on all composites. Concentration of these impacts ranged from two to seven impacts per 25 cm^2 (two to five impacts per sample). These impacts appear to fit generally into two categories; one is a penetrating impact which produces craters and another is a non-penetrating impact which leaves a wetting residue. Impacts can be easily seen on the aluminized composites, but are more difficult to find on the carbon fiber composites. Analyses of several of the non-penetrating impact residues show potassium, phosphorous, sulfur, and calcium to be the major constituents; the most probable source has been identified as spacecraft fluid dumps. No single impact larger than 1 millimeter was observed and areal coverage of non-penetrating impacts exceeded that of

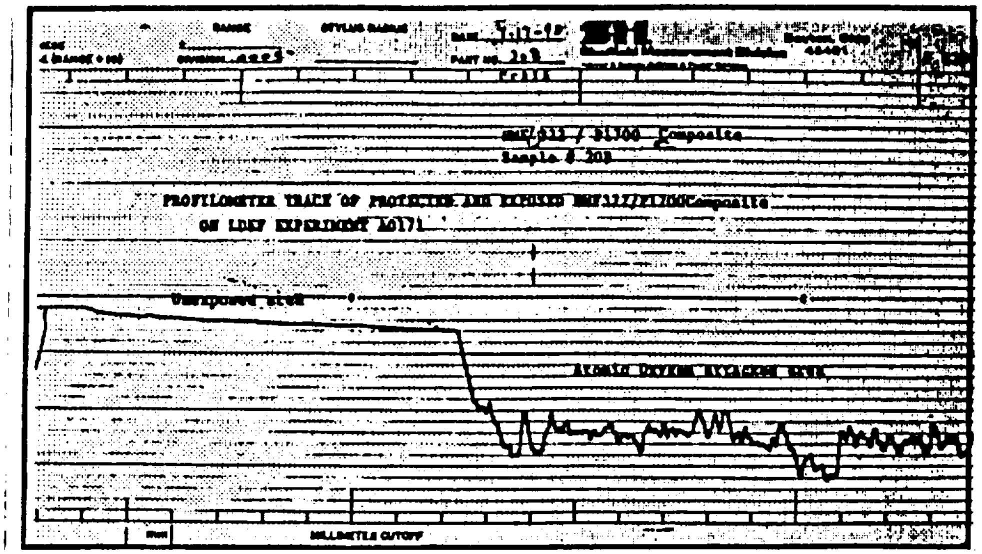


Figure 6. Profilometer trace of carbon fiber reinforced composite.

craters. Fiber breakage was evident in the crater impacts and would be expected to influence the tensile strength values generated for these samples.

Figure 8 reproduces SEM photographs of surface damage. In Figure 8, the upper left panel shows atomic oxygen erosion, texturing, ashing, and carbon fiber direction; the upper right panel shows peak type surface structure and ash; the middle left panel shows mesa formation, also on a carbon fiber composite. The middle right panel shows an impact crater with broken carbon fibers. The bottom panels show impact damage on S-glass composites covered with aluminum tape; on the left is a cratered impact, on the right splattered contamination.

Mechanical Effects

Two specimens from each of the five groups of carbon reinforced composites were tensile tested and compared with four control specimens. Observed changes are presumably a result of continued curing under UV and/or electron bombardment.

The Group 1 (polysulfone matrix) samples show uniform weakening.

The epoxy matrix composites show embrittlement with a general increase in Young's modulus; the modulus increased after LEO exposure in three out of four cases and, in the fourth case, showed a minor decrease in the face of a 21% decrease in ultimate stress.

Changes in ultimate stress of the epoxy based composites are anomolous and unexplained at this time. Ultimate stress dropped significantly in two cases (HMS-0° fiber and P75S-90° fiber) and

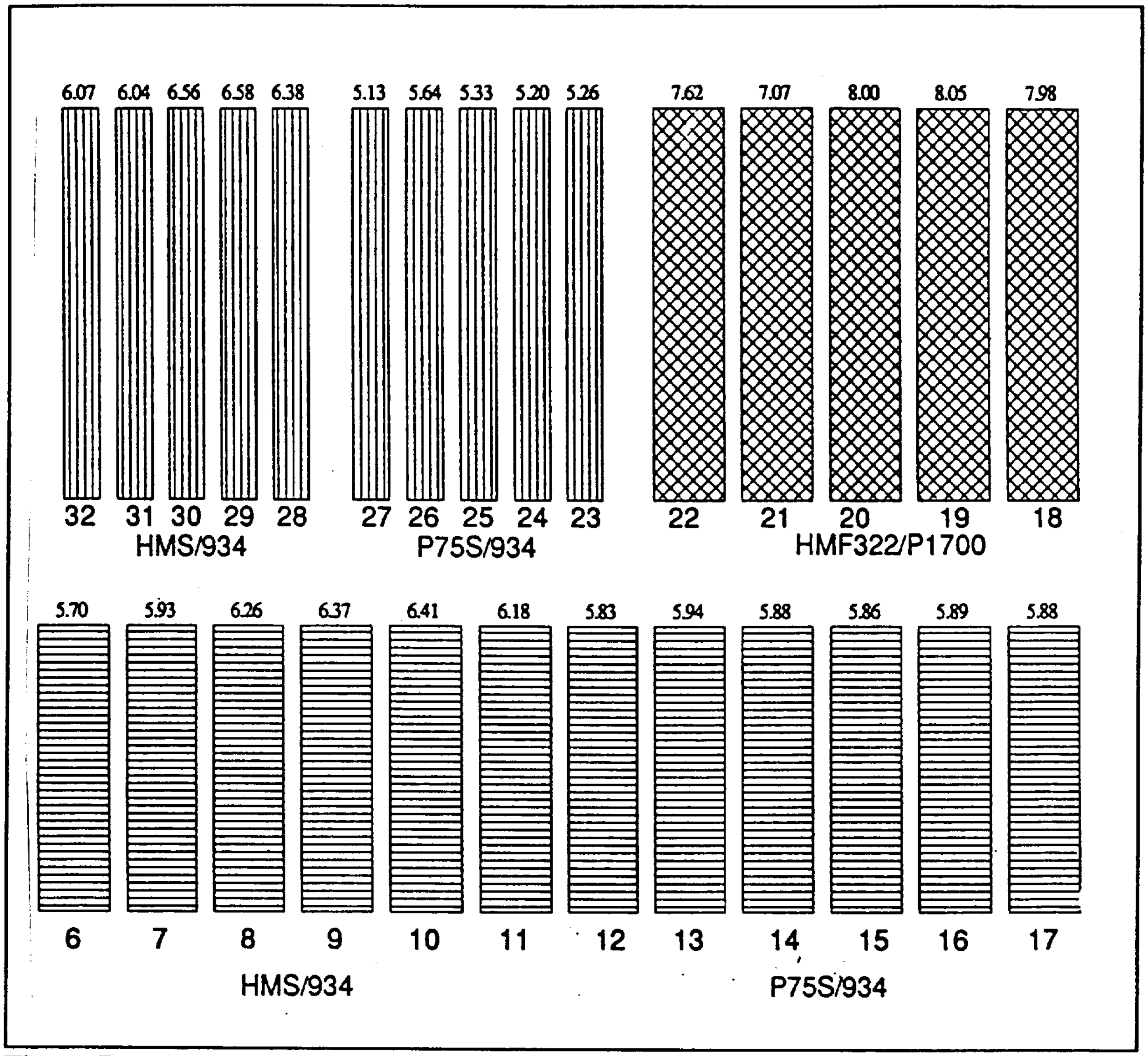


Figure 7. Layout of Plate II composites. Mass loss (%) and weave directions are shown.

remained essentially unchanged for two cases (HMS-90° and P75S-0°). Intuitively, one expects that changes in ultimate stress would be matrix dependent, but they seem to depend neither on weave, nor fiber type, nor matrix composition.

However, only two two samples of each flight group were tested, and testing was very difficult on the weakened 90° samples; even the control samples have little cross grain tensile strength. It is thus possible that this anomaly, if it is real, is an artifact of measurement statistics.

Table V lists the relevant data.

Table IV SURFACE EROSION OF COMPOSITES ON AO171

Group	Composite Material	Weave	Mass Loss (mg/cm^2)	Mean Thickness Loss (mils)	Atomic Oxygen Activity (10^{-24} cm/atom)
1[a,b,c]	HMF 322/P1700	±45°	18.46	4.7 - 11.5	1.9 - 4.6
2[a,b,d]	HMS/934	0°	11.79	2.5	1.0
3[a,b]	HMS/934	90°	11.31	2.7	1.1
4[a,b]	P75S/934	90°	11.27	2.7	1.1
5[a,b]	P75S/934	0°	10.29	2.8	1.1
6[b,c,f]	S-901/epoxy	0°&90°	2.40	0.36	0.14
7[g]	Al Taped S-901/epoxy	0°&90°	0.59	-	-

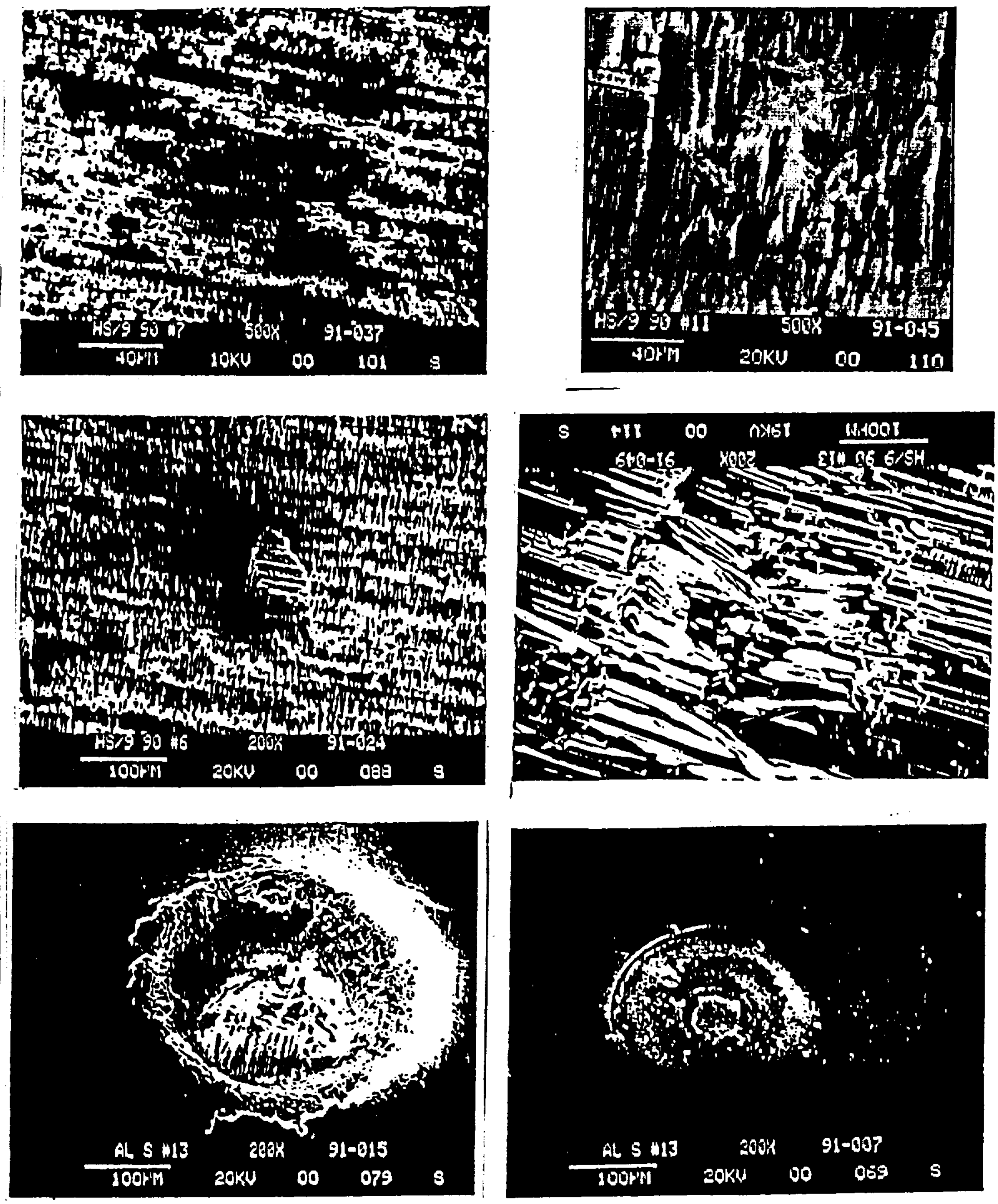
Notes:

(a) Darker and more diffuse than controls.
(b) Fibers evident.
(c) Matrix erosion much greater than fiber erosion.
(d) Average of rates from two ends of sample; contamination likely on forward end.
(e) Much darker than controls; probably results from UV irradiation.
(f) Fibers uneroded and became protective after initial matrix loss.
(g) Tape corrugated; probably related to many cycles of thermally induced stress.

CONCLUSIONS

Long duration mission spacecraft in LEO will require protection from radiation, atomic oxygen, and penetrating impacts for any of its light weight composites. To use thicker structures for reducing sensitivity to damage is not practical for mass and size considerations, and assumes

that the numerous small penetrating impacts do not influence bulk properties; this assumption has yet to be investigated, and may not be the case. Protective coatings for thinner composites, like the aluminized tape used on this experiment to protect fiberglass composites, must be fully



Upper left: oxygen erosion with texturing, ashing and fiber direction. Upper right: peak type structure and ashing. Middle left: mesa formation. Middle right: broken carbon fibers. Bottom left: impact crater on Al taape. Bottom Right: splatter.

Figure 8. SEM photographs of composite samples showing various damage mechanisms.

Table V TENSILE PROPERTIES OF COMPOSITES.

Composite Samples Tested		Ultimate Stress[a] (Kpsi)	Ultimate Strain[a] (%)	Young's Modulus[a] (10^6 psi)	Poisson's Ratio[a]
Group 1 HMF 322 P1700 ±45° Samples 18 & 19	Controls	23.2±9.7%	Note (b)	1.97±5.3%	Note (b)
	Flight	20.2±3.6%	Note (b)	1.71±0.83%	Note (b)
	Change	-13.0%	-	-13.2%	-
Group 2 HMS/934 ±0° Samples 29 & 30	Controls	161±7.2%	0.52±8.5%	28.5±4.1%	0.24±4.9%
	Flight	128±17.2%	0.33±22.3%	28.0±2.3%	0.26±5.9%
	Change	-20.8%	-35.4%	-1.75%	+8.1%
Group 3 HMS/934 90° Samples 8 & 9	Controls	3.29±9.7%	0.29±10.3%	1.13±4.7%	-
	Flight	3.3±17.3%	0.29±13.2%	1.16±3.7%	-
	Change	+1.3%	-1.7%	+2.7%	-
Group 4 P75S/934 90° Samples 14 & 15	Controls	2.69±7.8%	0.28±8.9%	0.950±2.8%	-
	Flight	1.8±14.8%	0.18±14.1%	0.981±0.9%	-
	Change	-34.4%	-36.2%	+3.3%	-
Group 5 P75S/934 0° Samples 23 & 24	Controls	95±13.6%	0.31±NA[c]	32.6±5.6%	0.30±NA
	Flight	97±30.6%	NA[c]	35.8±8.1%	0.33±6.9%
	Change	+1.9%	NA[c]	+9.8%	+11.1%

Notes:

(a) All uncertainties are standard deviations.
(b) Elongation of ≈7% was too large to permit measurement of ultimate strain.
(c) NA - not available; ultimate strain measurements made on one control sample.

characterized to understand the subtle changes experienced. This is necessary to provide confidence that the functional integrity of both the coating and composite will be maintained for long durations under space environmental conditions.

LDEF type missions for both shorter and longer exposures are the cost effective way to reach the goal of functional integrity at optimum cost. They would provide a means to gain new knowledge on space aging of materials and confidence in improving and interpreting ground based simulations of the space environment.