NASA Technical Memorandum

NASA TM-86463

(NASA-TM-86463) LEO ATOMIC OXYGEN EFFECTS ON SPACECRAFT MATERIALS: STS-5 RESULTS (NASA) 22 p HC A02/MF A01 CSCL 11G

N84-34566

Unclas
G3/23 24072

LEO ATOMIC OXYGEN EFFECTS ON SPACECRAFT MATERIALS — STS-5 RESULTS

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August 1984

National Aeronautics and
Space Administration

George C. Marshall Space Flight Center

MSFC - Form 3190 (Rev. May 1983)

TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO. NASA TM-86463	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.
4. TITLE AND SUBTITLE LEO Atomic Oxygen Effects on Spacecraft Materials — STS-5 Results		5. REPORT DATE August 1984 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Ann F. Whitaker		8. PERFORMING ORGANIZATION REPORT #
9. PERFORMING ORGANIZATION NAME AND ADDRESS George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812		10. WORK UNIT NO. 11. CONTRACT OR GRANT NO. 13. TYPE OF REPORT & PERIOD COVERED
12. SPONSORING AGENCY NAME AND ADDRESS National Aeronautics and Space Administration Washington, D.C. 20546		Technical Memorandum 14. SPONSORING AGENCY CODE
15. SUPPLEMENTARY NOTES Prepared by Physical Sciences Branch, Materials and Processes Laboratory.		

16. ABSTRACT

Effects of LEO atomic oxygen have been measured on a variety of spacecraft materials which obtained exposure on STS-5. Material degradation dependency on temperature was found in one material. Of the five paints flown, only S13GLO was unaffected. Generally, the glossy paints became Lambertian and the diffuse coatings improved. Scanning electron microscope examinations indicated removal of urethane and epoxy paint binder materials. Reaction products were evident on the surfaces of Z302 paint and Mylar. Thin films showed thickness losses ranging from negligible loss in Teflon to considerable loss in Mylar and Kapton. Glossy films such as black Kapton and white Tedlar became diffuse. Kevlar 29 rope lost tensile strength and silver solar cell interconnect material oxidized. Oxidation on the backside of an elevated silver specimen indicated that reflections of oxygen atoms were occurring and that reflecting surfaces, probably Kapton, were not fully accommodating the incident atoms.

17. KEY WORDS Atomic oxygen effects Spacecraft materials		18. DISTRIBUTION STATEMENT Unclassified-Unlimited	
19. SECURITY CLASSIF. (of this report) Unclassified	20. SECURITY CLASSIF. (of this page) Unclassified	21. NO. OF PAGES 20	22. PRICE NTIS

MSFC - Form 3292 (May 1969)

For sale by National Technical Information Service, Springfield, Virginia 22151

ACKNOWLEDGMENTS

This report presents the work of many people and the author wishes to acknowledge their efforts. Messrs. J. E. Coston and L. C. Wright produced the SEM photographs, R. J. Harwell and B. G. Kennedy prepared paints and provided optical property data, D. B. Griner and J. M. Carter provided BRDF measurements and analyses, L. M. Thompson made tensile tests, and Dr. P. N. Peters evaluated the silver interconnects. Mr. J. L. T. Ormsby and Ms. S. A. Little made mass loss measurements. The typing of the manuscript by Ms. Jewell Cargile is especially appreciated. Dr. Alan Biddle did the early coordination on this experiment. Mr. W. R. DeWeese for metallurgically mounting and photographing aluminized kapton.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

ORIGINAL PAGE IS OF POOR QUALITY

TECHNICAL MEMORANDUM

LEO ATOMIC OXYGEN EFFECTS ON SPACECRAFT MATERIALS — STS-5 RESULTS

INTRODUCTION

The STS-5 experiment, "Evaluation of Oxygen Interaction with Materials," was developed to obtain quantitative data on the reaction rates of spacecraft materials and to assess the materials degradation dependency on temperature. Mass loss and rapid aging observed respectively on thin organic films and paints in the Shuttle cargo bay on earlier STS flights clearly indicated a need for this data. Degradation effects in these materials have been attributed to reaction with atomic oxygen which is a highly oxidizing agent and the predominant species in the 200 to 600 km LEO altitude range. The duration of exposure of the STS-5 experiment during active thermal control was planned so as to obtain incident atomic oxygen fluence on materials similar to that experienced in earlier flights. A total fluence of atomic oxygen atoms of 9.9×10^{19} atoms/cm^2 was calculated [2] to be incident on the materials during this flight.

The flight configuration of STS-5 material specimens is shown in Figure 1 on one of the temperature controlled plates. Those materials identified are included in this analysis. The material specimens configured in 2.54 cm (1 in.) wide strips were held in thermal contact with the temperature controlled plate surfaces by a series of tension springs. Two identical trays each with three temperature controlled plates comprised the major portion of this experiment [1,2].

The analyses plan for the spacecraft materials flown is described in Table 1. The number of specimens flown and evaluated is listed in parentheses beside each identified material. Those materials listed as having one or two specimens were those shared by other investigators. In several instances, analyses were tailored to a current critical application of material. For example, the light scattering characteristics of Z302 paint and the retroreflector properties of 3M-7610 are critical applications and require examination through bidirectional reflectance distribution function (BRDF) analyses. Mass loss was determined on thin film and cable materials. The thickness and massiveness of the paint samples/substrates and the irregular shape and partial exposure of the silver interconnects prevented attempts at their mass loss/gain determinations. Microscopic examinations were made of all specimens. Other characterizations including optical properties, tensile strength, X-ray diffraction, EDAX and photomicroscopy served as the primary analysis or were made in support of a particular effect observed in the material.

Visually, surface changes noted ranged from a reduction of glossiness in the paints and thin films to oxide scale accumulation on the silver. No material transfer was obvious. The loose reaction products seen on some of the material surfaces constitute a particulate contamination hazard if they are disturbed. The property changes observed and determined through analyses in these materials can be significant depending on their optical, mechanical and/or electrical application.

DATA PRESENTATION AND ANALYSES

Paint

The MSFC furnished paints flown on the STS-5 atomic oxygen experiment included two white paints, S13GLO and Chemglaze A276, and three black paints, Chemglaze Z302, Z306 and 3M 401-C10. The Z302 and A276 are glossy, and the Z306 and 401-C10 are diffuse coatings. These paints were applied to aluminum foil strips which were held in good thermal contact against the flight heater plates. During the active portion of the experiment the paint strips were maintained at the temperatures (75°F, 150°F and 250°F) of the respective heater plates. A small area on each paint specimen was masked off with aluminum foil prior to flight for the purpose of determining the erosion depth of the paint after flight. However, it was found that the paints which were affected generally were preferentially attacked so that the remaining unaffected surface particles interfered with attempts to measure paint recession.

No optical property degradation dependency on temperature was noted so all of the optical data was averaged together. A summary of optical property data, solar absorptivity (α_S) and total solar reflectance (R_T) with diffuse (R_D) and specular (R_S) components, where applicable, is shown in Table 2. Values shown with 1σ variations are the average for five specimens. Solar reflectance was measured from 0.337 to 2.789 μm and is represented by the average of twenty values over that wavelength region. Solar absorptivity was calculated from reflectance. No change was noted in emissivity for any of the paints. Optical property degradation for the affected paints was fairly uniform over the total wavelength range. Bidirectional Reflectance Distribution Function data were also generated on all the paints.

Solar absorptivity and scanning electron microscope (SEM) photographs confirm no surface changes in the S13GLO paint. The RTV 602 base material of this paint appears to be relatively resistant to atomic oxygen attack. Generally the glossy surfaces, A276 and Z302, became Lambertian upon exposure with the small specular component of A276 reflectance decreasing by 53 percent and the small diffuse component of the Z302 reflectance increasing by 200+ percent. A 1+ percent increase in α_S was noted for Z302, and no change was noted within measurement error and control sample-to-sample variation for α_S in the A276. Of particular interest is the visible light scatter data for Z302. This data is provided for normal incidence at 632.8 nm in Figure 2 for an exposed and a control specimen in the BRDF format described in Reference 3. The plots provide normalized measured scatter with its angular dependence at 632.8 nm for the two specimens. It can be seen that the exposed Z302 is more uniformly diffuse and that its specular reflectance has diminished by about a factor of three.

SEM photographs for the exposed and unexposed A276 and Z302 are shown respectively in Figures 3 and 4. The A276 developed a porous surface upon exposure, and loose clusters of surface particles less than 2 μm in diameter could be removed by gently rubbing. The atomic oxygen appeared to be reacting with the A276 polyurethane binder leaving paint pigment particles on the surface. The exposed Z302 surface showed patterned reaction product debris on the surface.

The diffuse coatings, Z306 and 401-C10, improved with exposure. Small increases in α_S values were noted for the flight specimens. SEM photographs in

Figures 5 and 6 indicate preferential attack of the exposed paint surfaces with the binder materials being removed. Clusters of particles about 4 μm in diameter remained on the porous Z306 surface while the binder connecting the surface micro-balloons in the 401-C10 was removed.

Silver

Two silver solar cell interconnects were flown on the temperature controlled heater plates. The interconnects were Teflon taped to Kapton H strips which were held in thermal contact with the 75°F and 150°F heater plates. A portion of the interconnect on the 75°F heater plate was lost prior to flight leaving one edge slightly elevated above its Kapton substrate. The poor thermal contact of the interconnect probably prevented it from being maintained at 75°F during the active portion of the experiment. The flown interconnects, Figure 7, were discolored and scaly and possessed none of their preflight high reflectivity. Dark scale was observed on the front sides (facing space) of both interconnects and the back side (facing Kapton) of the elevated interconnect. The scale material was identified tentatively as an oxide of silver. No sulfides or other heavy element compounds were found in the silver. Scales which debonded but remained in place on the silver impeded the reaction process. However, when they flaked off a fresh surface became available for renewed reactions. Front side scale thicknesses were determined to be ∿2μm on the 150°F plate interconnect and ∿1μm on the 75°F plate interconnect. The thicker scale on the hotter interconnect suggests that a temperature dependent diffusion process is involved in the scale formation. The scale present on the back side of the elevated interconnect indicates that reflections of atomic oxygen atoms were occurring and that the reflecting surfaces, probably Kapton, were not fully accommodating the incident atomic oxygen atoms. This information was summarized from a draft report [4] prepared on the silver analyses.

Silver foils flown, Figure 8, at 200X magnification, show brownish patches and preferential brownish striations on the exposed sides. Further analysis of this material is required.

Thin Films

Mass loss analyses of thin film materials flown on the STS-5 atomic oxygen experiment were made by both MSFC and JSC and the data combined. No degradation dependency on temperature was noted so the data on the films at various temperatures were averaged together. The detailed mass loss determinations on these films are to be reported by JSC. Weight differences from control to exposed specimens were attributed to mass losses in the flight specimens as a result of atomic oxygen exposure. Results were reported as thickness losses. Kapton H and Mylar showed the greatest thickness losses up to ∿0.09 to 0.10 mil with these losses characteristic of the initial thickness of these films - thicker (2 mil Kapton) and thinner (1/2 mil Mylar) films showed the greatest apparent thickness reductions. It has been suggested that possible processing induced variations in the surface density of the different thicknesses of these films are responsible for this response to the atomic oxygen environment. Figure 9 shows a cross section of aluminized Kapton H with an exposed and protected Kapton region. The exposed region thickness recession is ∿0.063 mil. Laboratory evaluations of 2 mil Kapton H in a thermal oxygen environment show an apparent related response with the greater mass loss rate occurring within 0.05 mil of the surface than within the bulk of the film.

White Tedlar showed thickness losses of ∿0.02 mil and black Kapton of ∿0.05 mil. An SEM photograph, Figure 10, of exposed black Kapton reveals a roughened microscopic surface as a result of the reaction with atomic oxygen. Aluminum of approximately 150 Å appeared to provide Kapton protection to atomic oxygen but could not be fully confirmed by SEM examination. Mass loss was negligible on the one specimen of FEP Teflon examined. SEM photographs of 3M-7610 retroreflector tapes were inconclusive in providing evidence of loss of material as a result of exposure.

Optical properties of selected thin films are shown in Table 3. The glossy films, black Kapton and white Tedlar, tended to become Lambertian upon exposure. A white reaction product was evident on the Mylar and combined transmission and reflectance measurements at 30° were made with and without the surface product to determine its relative effective magnitude on the optical properties of Mylar. A 2 percent decrease in the measured value was noted on the flight specimen. Limited Mylar specimens prevented attempts to identify the surface product. The 3M-7610 retroreflector showed no changes in reflectance at 632.8 nm.

Kevlar 29

Braided Kevlar 29 rope, ∿0.32 cm (1/8 in.) in diameter by 23 cm (9 in.) long, was flown on each of the temperature controlled plates on one tray. Evaluation of these braided ropes was made based on changes in mass per length and comparative tensile strength. A mass per length value of 19.93 mg/cm (50.61 mg/in.) was established for the flight material in the as-received condition. It was then subjected to a thermal vacuum bake for 24 hr at 250°F and $\sim 1.3 \times 10^{-4}$ pascal (1×10^{-6} torr) prior to flight. Control material was subjected to a similar bakeout, weighed immediately afterwards and several days later after it regained desorbed moisture. Upon retrieval mass per length values were determined for the flown specimens. These values were then compared to the control values to establish the mass loss per length attributable to the atomic oxygen environment. Mass loss per length due to thermal vacuum environment alone was 4.5 percent with 1.5 percent being desorbed moisture. Mass loss per length attributable to atomic oxygen ranged from 0.3 to 3.3 percent for the flight specimens with no correlation to temperature being apparent. A portion of the 75°F plate specimen appeared to have a relaxed weave. This factor could possibly account for more mass loss on this specimen since more fiber area was exposed to the atomic oxygen environment. Comparative tensile strength tests on these short specimens revealed a range of strength reductions up to 41 percent. No clear break was apparent in the braids and no strength correlation could be made to temperature or mass loss per length. This data is summarized in Table 4. An SEM photograph of an exposed Kevlar 29 fiber, Figure 11, shows a region of apparent chemical attack.

CONCLUSIONS

Significant property degradation was measured in almost all of the exposed materials. S13GLO paint was the exception with no changes detectable in the exposed material. Attack of polyurethane and epoxy binders was evident in the SEM photographs of affected paints. Generally the specular coatings became diffuse and the diffuse coatings improved. Atomic oxygen exposure can have a severe impact, for example, on optical systems that depend on a coating's specular reflectance for stray light suppression. Solar absorptivity increased in these paints as a result of the voids left on the surface from binder erosion. Loose particles which remained on the affected surfaces can yield particulate contamination if disturbed.

Silver oxidized in this environment and showed some dependency on temperature in the thickness of oxide developed. The oxide layer provided little surface protection since it tended to flake off. If this oxidation process continued at a linear rate then silver interconnected solar cells would develop high series resistances and eventually open circuits. It is expected that the stress condition along with other processing parameters may influence the rate of oxidation in silver.

The principal effect determined on the thin films was mass loss. The mass loss observed for Kapton has a substantial impact since Kapton serves as the structural blanket in all state-of-the-art solar arrays. Optically the glossy films such as black Kapton and white Tedlar became diffuse. Black Kapton and white Tedlar are used as light shields with stray light requirements and it is important that they not be eroded through or become completely diffuse.

Mass loss and tensile strength reduction were noted in Kevlar 29 rope. Many questions remain concerning the response of this material since no correlations were made relating mass loss, tensile strength and temperature dependency of these properties. The response of this material to the atomic oxygen environment requires further investigation.

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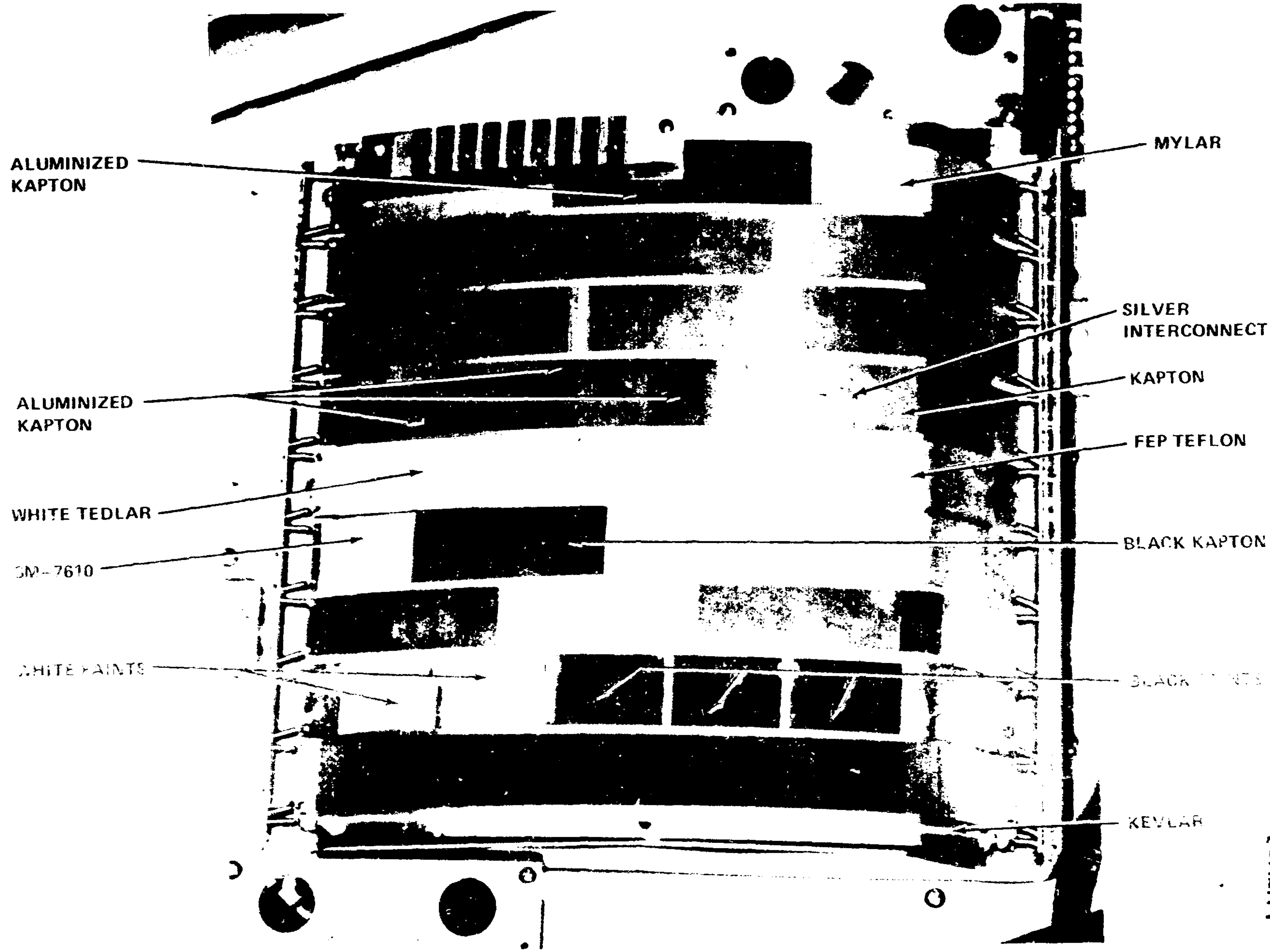

Figure 1. STS-5 flight configuration of temperature controlled materials specimens.

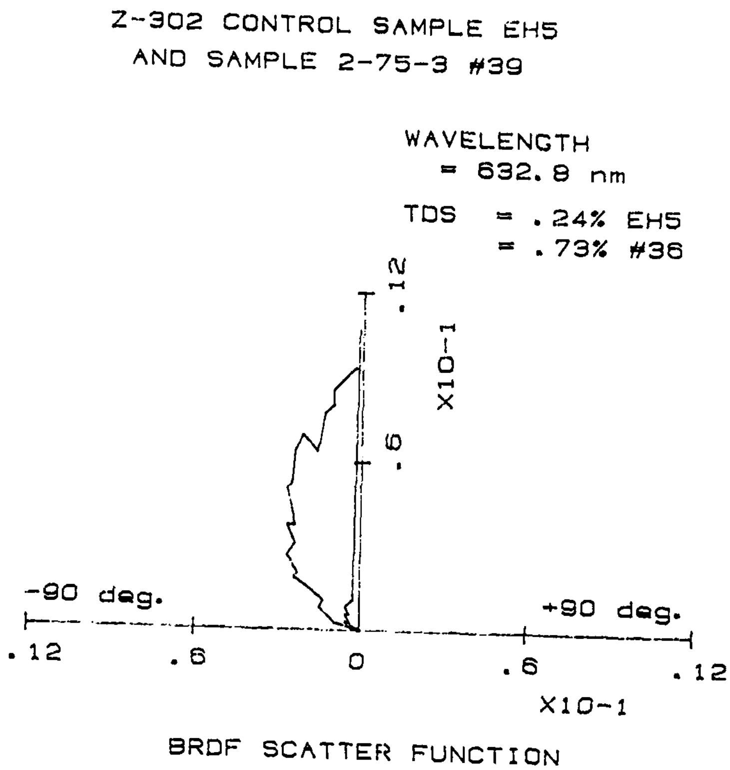

Figure 2. Measured scatter for exposed and control specimens of Z302 paint.

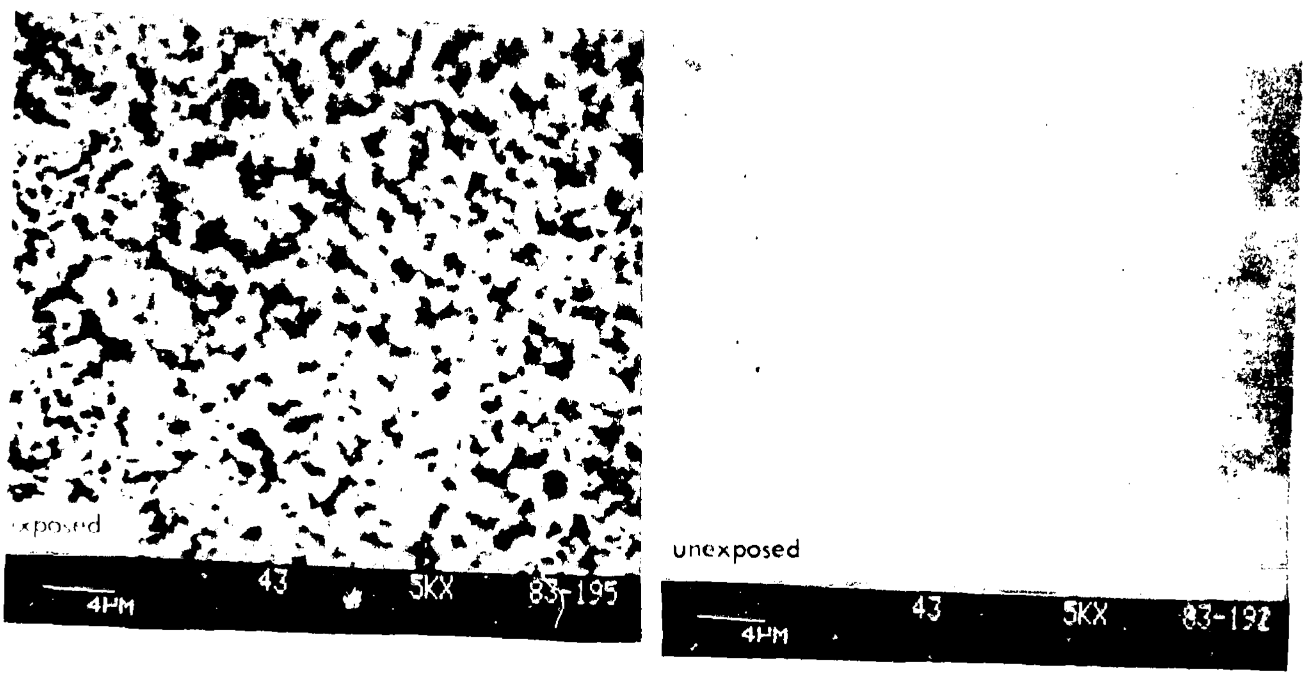

Figure 3. SEM photograph of exposed and unexposed A276 paint.

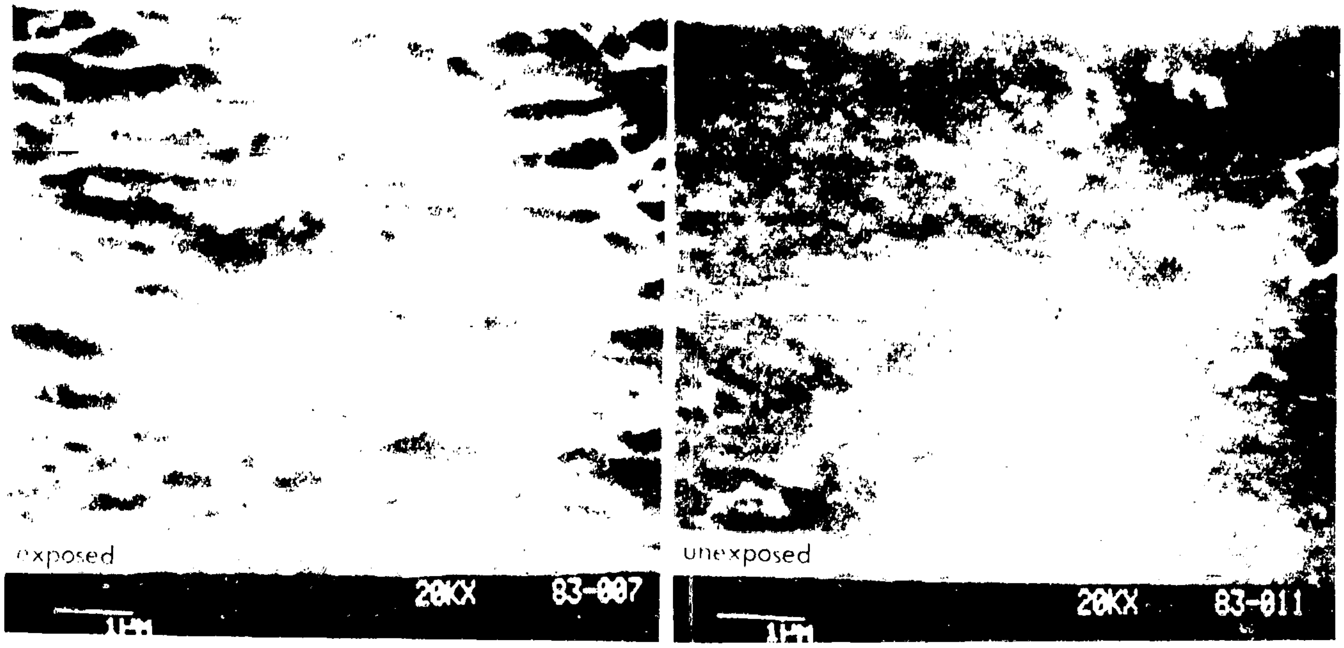

Figure 4. SEM photograph of exposed and unexposed Z302 paint.

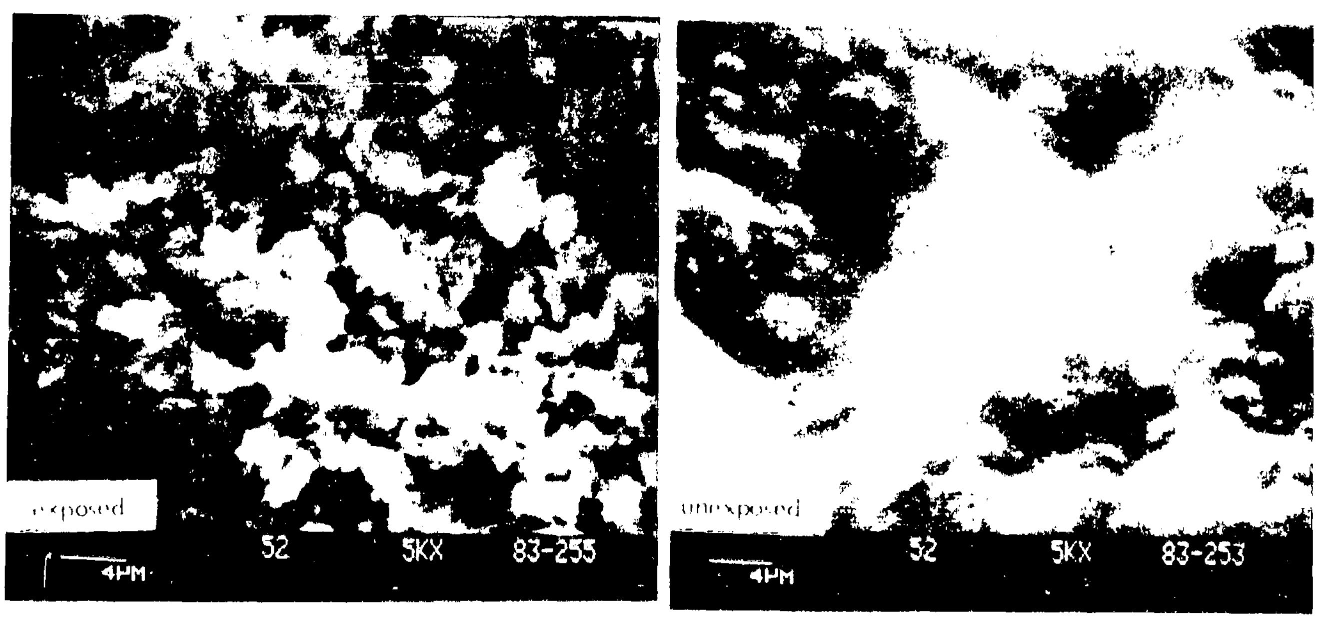

Figure 5. SEM photograph of exposed and unexposed Z306 paint.

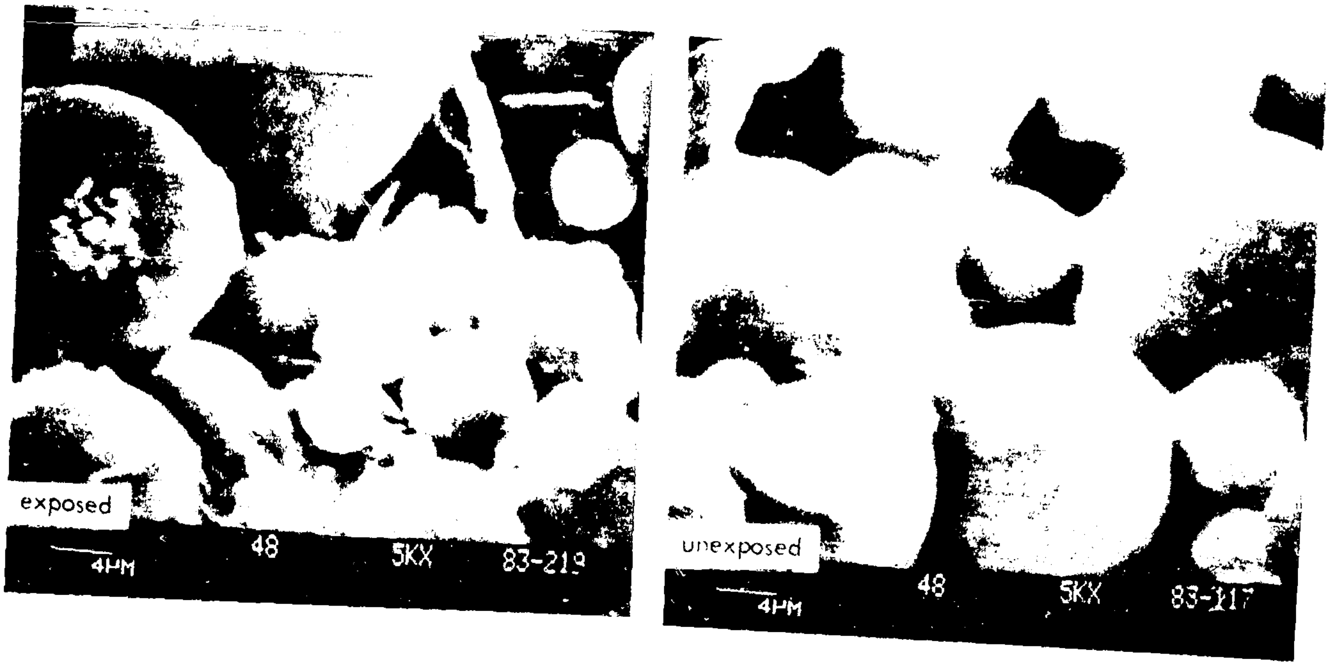

Figure 6. SEM photograph of exposed and unexposed 401-C10 paint.

Figure 7. A section of exposed STS-5 silver interconnect.

Figure 8. Silver foil following exposure on STS-5.

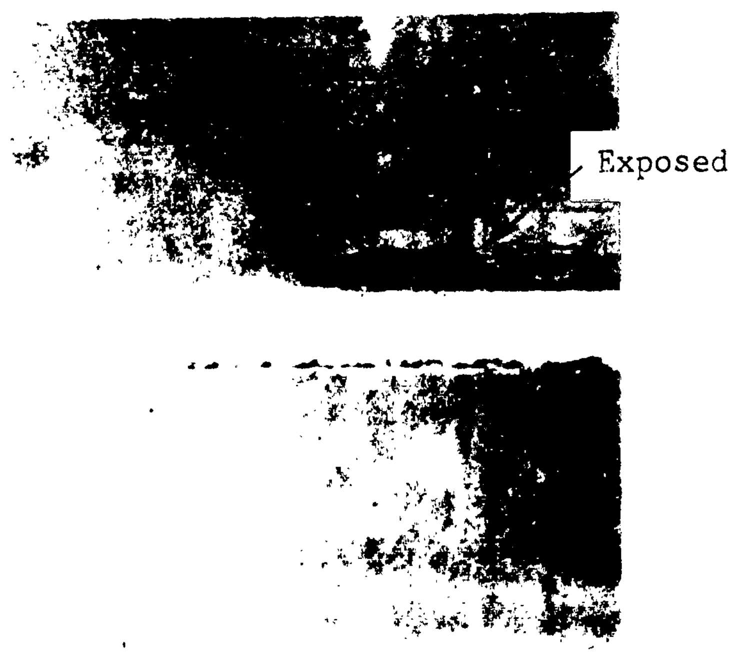

Figure 9. Cross section of aluminized Kapton at 400X showing exposed and protected Kapton region.

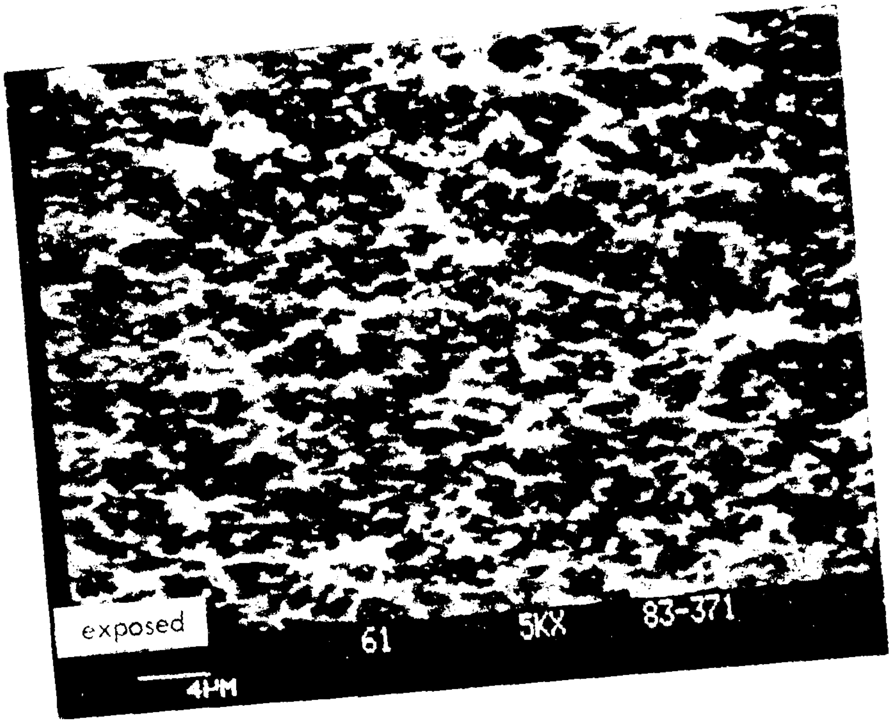

Figure 10. Black Kapton following exposure on STS-5.

Figure 11. SEM photograph of exposed Kevlar 29 fiber.

TABLE 1. EVALUATION MATRIX FOR STS-5 MATERIALS

Paints	Metal	Thin Films	Cable
S13GLO (5)	Silver Inter-connects (2)	Kapton H (3)	Kevlar 29 (3)
Z306 (5)	Silver Foil (2)	Mylar (2)	
Z302 (5)		Black Kapton (5)	
A276 (5)		White Tedlar (1)	
401-C10 (5)		Aluminized Kapton (1) (50A, 100A, 150A, 2000 A)	
		FEP Teflon (2)	
		3M-7610 (6)	
↓	↓	↓	↓
Analyses	**Analyses**	**Analyses**	**Analysis**
Optical Properties α_S, R_T, R_S, R_D BDRF	SEM	Mass Loss	Mass Loss/Length
SEM	X-Ray Diffraction	Optical Properties α_S, T, R, BDRF	Comparative Tensile
	EDAX	SEM	SEM
	Photomicroscopy		

TABLE 2. OPTICAL PROPERTY DATA ON STS-5 PAINTS

Specimen ID/Comments	S13GLO	Z306	401-C10	A276				Z302			
	α_s	α_s	α_s	α_s	R_t(%)	R_d(%)	R_s(%)	α_s	R_t(%)	R_d(%)	R_s(%)
Flight Specimens, Avg Value for all Temps.	0.186 ±0.003	0.988 ±0.001	0.981 ±0.001	0.222 ±0.005	77.8 ±0.5	76.5 ±0.3	1.3 ±0.6	0.967 ±0.004	3.4 ±0.4	1.9 ±0.7	1.5 ±0.4
Control Value	0.184 ±0.003	0.954 ±0.001	0.976 ±0.001	0.227 ±0.002	77.3 ±0.2	74.6 ±0.6	2.7 ±0.5	0.956 ±0.001	4.4 ±0.1	0.6 ±0.4	3.8 ±0.5
Comments on Exposure Effects	No Change	+3.6% in α_s more diffuse porous surface	+0.5% in α_s more diffuse	No Change	No Change	+2.5% in R_d	-53% in R_s	+1.1% in α_s	-22.7% in R_t	$+200^{+}$% in R_d	-60% in R_s
				More diffuse, porous surface, paint could be removed after exposure by gently rubbing.				Reaction products on surface.			

TABLE 3. OPTICAL PROPERTY DATA ON STS-5 THIN FILM MATERIALS

Spec. ID/ Comments	Al/Kapton (1/2 mil) α_S	Black Kapton (1 mil) α_S	Mylar (2 mil) $T+R_T$(%) @ 30°	Mylar (2 mil) T(%)	White Tedlar (1 mil) $T+R_T$(%) @ 30°	White Tedlar (1 mil) T(%)	3M-7610 R_D at 632.8 n.mi.
Flight specimens, average value for all temps	0.338	0.963 ±0.003	95.5 (96.2)*	79.0 (80.9)*	70.3	3.2	24.9 ±0.7
Control value	0.315	0.922	97.5 (97.5)*	81.4 (81.4)*	70.6	3.3	25.5

*Cleaned.

TABLE 4. PROPERTY DATA ON STS-5 KEVLAR 29 ROPE

Specimen No. Tray - Temp, °F	Reduction in Mass/Length, %				Reduction in Comparative Tensile Strength, %
	STS-5 Specimen After Flight	Control Immed. After TV Bake 24 hrs/250°F/10^{-3} torr	Control After 5 Days Amb. Temp/Press.	STS-5 Loss Att. to Atomic Oxygen	
1-75*	6.4	4.5	3.1	3.3	29
1-150	3.7	4.5	3.1	0.6	41
1-250	3.4	4.5	3.1	0.3	33

*About 2/3 of 23 cm length appeared to be a loose weave, ∿0.2 cm wider.

APPROVAL

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By Ann F. Whitaker

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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☆U.S. GOVERNMENT PRINTING OFFICE 1984—746 070/10034