NASA Technical Memorandum 104574

Four Space Application Material Coatings on the Long-Duration Exposure Flight

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Four Space Application Material Coatings on the Long-Duration Exposure Flight (LDEF)

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PREFACE

Four material coatings of different thicknesses were flown on the LDEF to determine their ability to perform in the harsh space environment. The coatings, located in the ram direction of the spacecraft, were exposed for 10 months to the low-Earth orbit (LEO) environment experienced by the LDEF at an orbit of 260 nautical miles. They consisted of: Indium Oxide (In_2O_3), Silicon Oxide (SiO_x), clear RTV silicone, and Silicone with Silicate-treated Zinc Oxide (ZnO). These coatings were flown to assess their behavior when exposed to atomic oxygen and to confirm their good radiative properties, stability, electrical conductivity, and resistance to UV exposure.

The flown samples were checked and compared with the reference unflown samples using high-magnification optical inspection, ESCA analysis, weight changes and dimensional changes. These comparisons indicated the following.

The 1000Å SiO_x coating eroded uniformly, with minor changes in its radiative properties. The 100Å of In_2O_3 coating eroded completely down to the Kapton® backing, with resultant losses of reflectance. The RTV-615 showed erosion, with carbon (C) content losses, while the Si remained constant, with a doubling of the oxygen (O) concentration. The RTV-615 silicone with K_2SiO_3-treated ZnO changed from flat to glossy white in appearance. It lost C, was etched, and increased its O content. The upper layers showed no remaining Zn or K. Losses of reflectance occurred within certain wavelength bands.

It was not possible to evaluate the experimental oxygen reaction rate using the calculated atomic oxygen fluence of 2.6×10^{20} atoms/cm^2 for the exposure of these coatings during the flight. The bakeout of the coatings was not carried out prior to the flight. Hence, the coating weight and dimensional losses included losses by outgassing products.

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1. INTRODUCTION

Four coating samples prepared by the Materials Branch at GSFC were flown on the Long Duration Exposure Flight (LDEF) spacecraft, launched in 1984. The samples were mounted with many others in the Experiment Environmental Control Canister (EECC). The canister was identified as Experiment No. S0010, and was located in Tray B9, which was situated at an angle of 8.1° from the ram vector, as shown in Figure 1. The assembly of the test specimens in the flight canister and in the control canister was managed by NASA's Langley Research Center (LaRC).

The flight canister, shown in Figure 2, was provided with a drawer that opened and closed on command to expose the samples to the space environment while in flight. The container provided a clean, low-pressure inert gas environment while closed. A timer opened and exposed the samples 1 month after launch, and remained open for 10 months, at which time the drawer returned to the closed position to protect the samples during the remainder of the mission. The hermeticity of the drawer and canister was reconfirmed on the LDEF return, some 5 1/2 years after launch.

Table 1 shows the environmental exposure conditions as reported in Reference 1. The atomic oxygen fluence for the spacecraft (particularly for Row 9) and other data on the space environment are shown in Figures 3 and 4, taken from Reference 2.

For the 10-month exposure at an altitude of 260 nautical miles, the oxygen fluence is estimated to have been (from Figure 3 of the above reference) 2.6×10^{20} atoms/cm^2. The UV radiation exposure was 126,000 hours, as indicated in Table 1. The other environmental parameters are given in Table 1.

The four samples were located on the tray in the same row that included samples from the GSFC Optics Branch. Those samples consisted of various metallic coatings such as Au (gold), Pt (platinum), Os (osmium), Ir (iridium), and Al (aluminum) with MgF (magnesium fluoride) and SiO_x (silicon oxide).

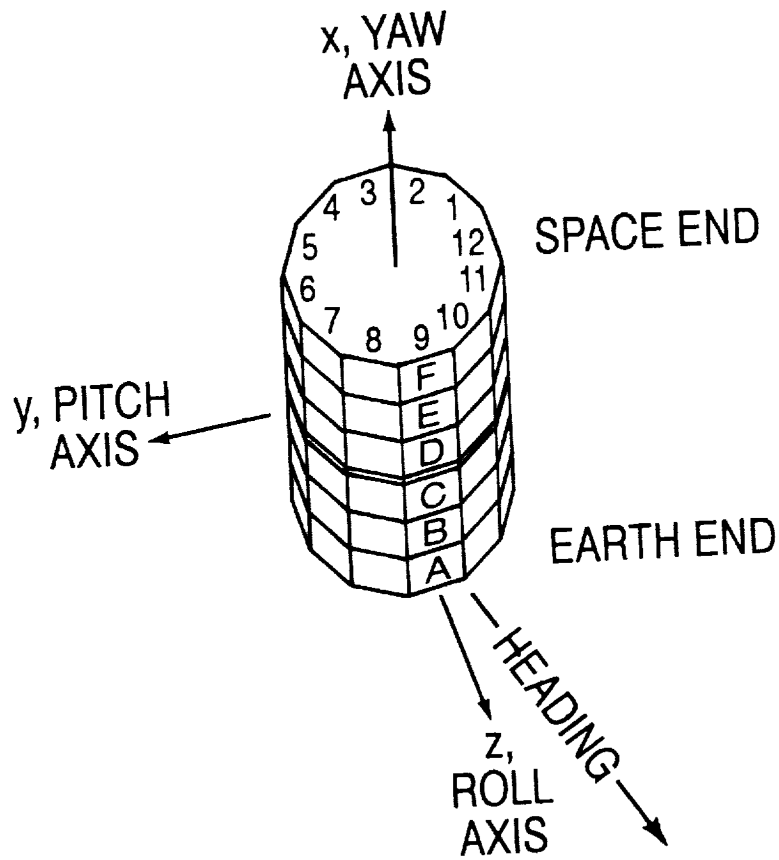


Figure 1. LDEF sketch and orbital configuration.

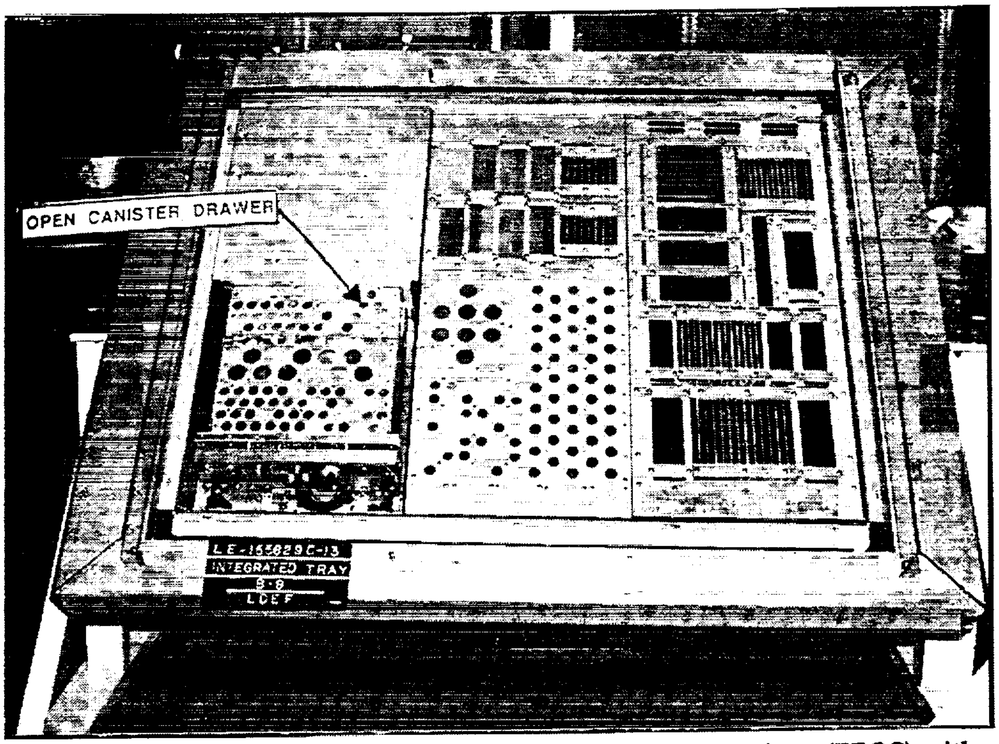


Figure 2. Photograph of experiment environmental control canister (EECC) with test specimen installed. (Photo L-83-10,250)

Table 1. Preliminary Environmental Exposure Conditions

Atomic Oxygen* 0 to 10^{22} atoms/cm^2 (wake to ram)	Vacuum $10^{-6} - 10^{-7}$ torr
UV radiation 100-400 nm; 16,000 hrs	Thermal cycles ~34,000 cycles: –20 to 190°F, ±20°
Particulate radiation e^- and p^+: 2.5×10^5 rad surface fluence Cosmic: <10 rads	Altitude 255–180 nautical miles
Micrometeoroid and debris 6000 particles from 0.1 mm to 2 mm	Orbital inclination 28.5°

*Updated value of 8.4×10^{21} as in Figure 4.

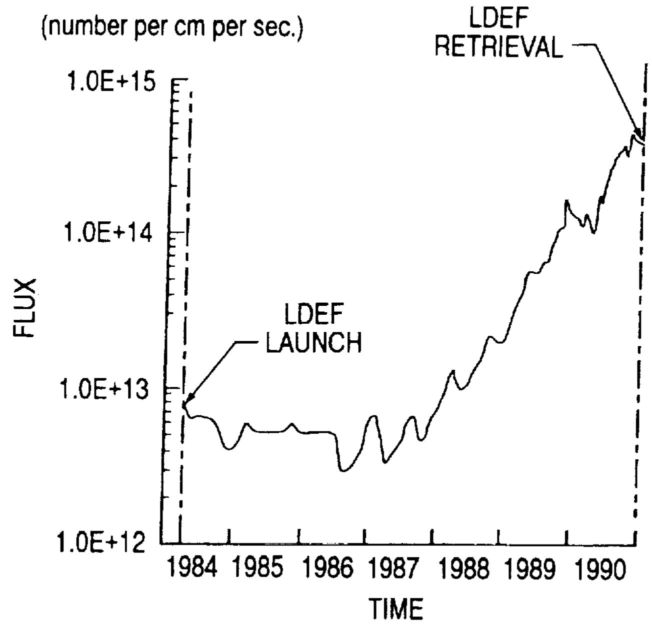
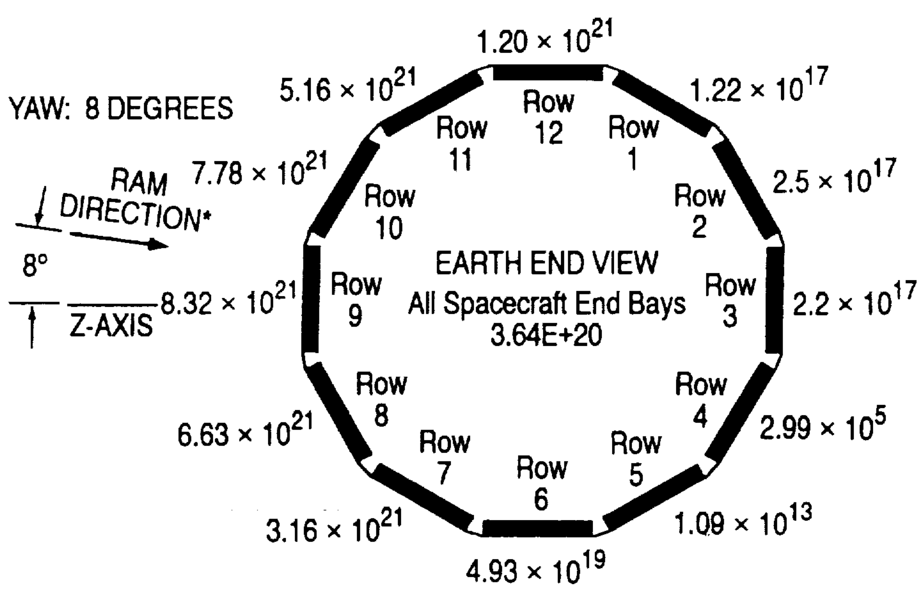


Figure 3. History of oxygen fluence on LDEF leading surfaces.



*RAM DIRECTION: 8.40E+21 atoms per sq cm

Figure 4. Calculated distribution of the total atomic oxygen fluence on each of the LDEF surfaces.

2. DESCRIPTION OF COATING SAMPLES

The four coating samples are shown schematically in Figure 5, indicating their compositions and the known dimensions. It is not known if, previous to the flight, those samples were baked out in vacuum to reduce their outgassing. We are assuming that they were not. The descriptions of the samples, the primary uses and advantages of the coatings, and available data on the samples follow.

Kapton®/VDA, 1000Å SiO_x (Sample #3)

This sample was composed of 1000 Å of SiO_x deposited on the vacuum-aluminized face of Kapton, which was attached to the aluminum support disk with 3M Corporation's Y-966 transfer adhesive. Data on the sample indicate that the weight of the assembled components was 4.345883 g and its total thickness was 0.1294 in. (0.3287 cm). The weight of the support disk was approximately 4.25987 g and its thickness was 0.1148 in. (0.2916 cm). No other data was given. The surface was described as "shiny metallized." This combination is often used as an environmental protective coating, is resistant to atomic oxygen exposure, and provides improved radiative properties after space environment exposure.

Kapton/VDA with 100 Å In_2O_3 (Sample #2)

This sample consisted of 100 Å of indium oxide deposited on Kapton. The Kapton was attached with its vacuum-deposited Al face to the aluminum support disk with 3M's Y-966 adhesive. The data sheet indicates that the assembled sample weight was 4.328355 g and its thickness was 0.1271 in. (0.3228 cm). The support disk weight was approximately 4.259878 g and its thickness was 0.1160 in. (0.2946 cm). The surface was described as "yellow," and "shiny." The indium oxide coating provides sufficient electrical conductivity, has little effect on substrate solar absorption and emittance, and remains stable during long exposure in space to UV radiation and particle bombardment.

RTV-615 Silicone on Aluminum (Sample #2C)

This sample consisted of devolatized General Electric Corporation (GE) RTV-615 two-part silicone with an A/B parts-by-weight mix ratio of 10/1, which was bonded to the aluminum disk via GE primer SS4155. The data describe it as a clear silicone. The total thickness of the assembly was 0.1253 in. (0.3183 cm). The weight of the support disk was 4.25987 g and its thickness was 0.1127 in. (0.2862 cm). No other description was given. This combination is an environmental protective coating used as a sealant and is particularly resistant to atomic oxygen.

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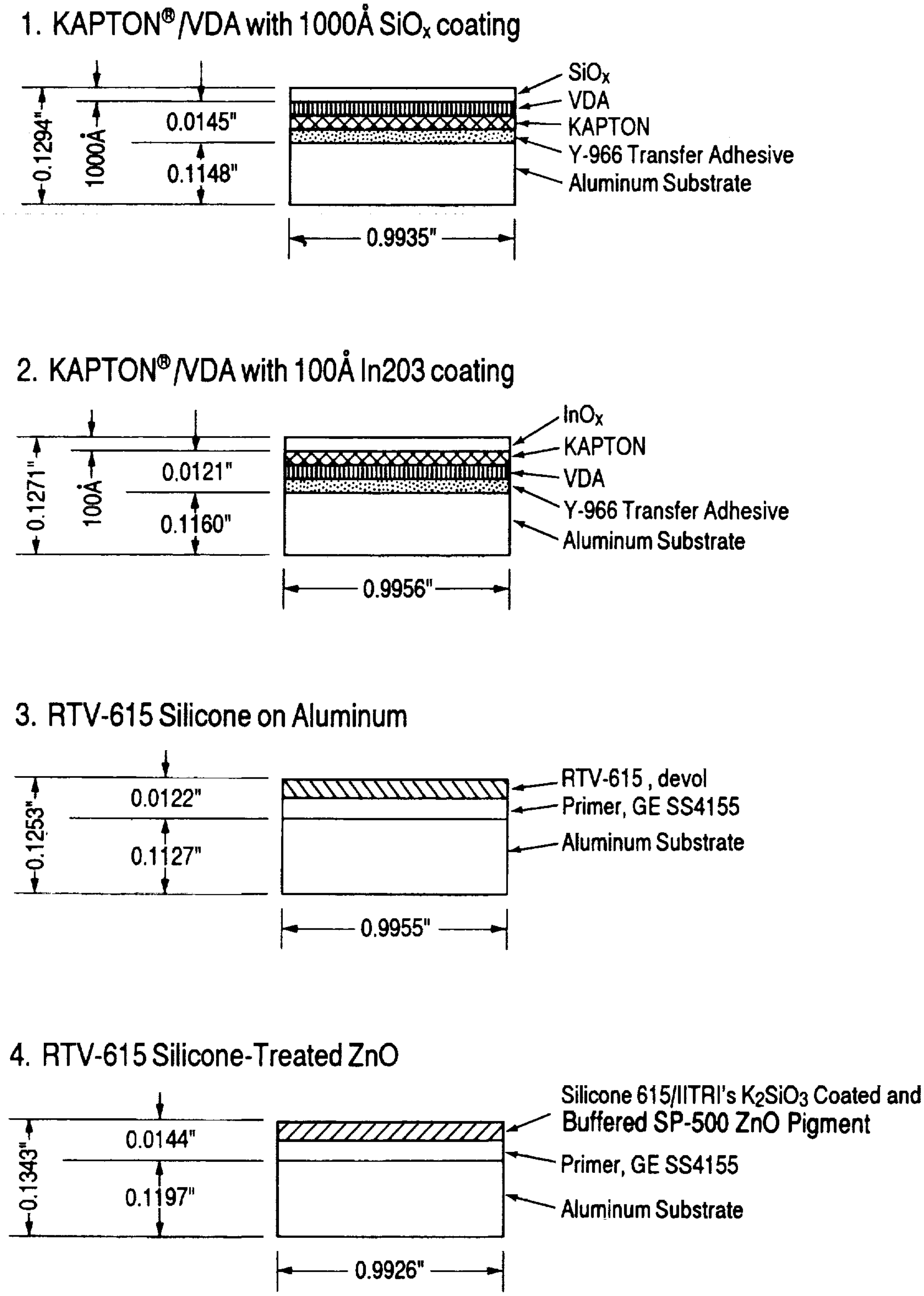

Figure 5. Side views of LDEF specimens (not drawn to scale).

RTV-615/Silicone-Treated ZnO (zinc oxide) (Sample #1A)

This sample consisted of GE's devolatized RTV-615 two-part silicone with 68% of IITRI's K_2SiO_3 coated and buffered SP-500 ZnO pigment. The RTV-615 silicone had an A/B parts-by-weight mix ratio of 10:1. The material was bonded to the aluminum disk via GE primer, SS4155. The total weight was 4.55060 g and the total thickness was 0.1343 in. (0.3411 cm). The weight of the support disk was 4.25987 g and its thickness was 0.1197 in. (0.3040 cm). No other data were given. This combination is a thermal control coating and is used as a white paint for spacecraft and other structures. It is resistant to UV radiation exposure.

3. DATA AND MEASUREMENTS TAKEN

The effect of the space environment on these samples is indicated by providing the following descriptive parameters.

- The sample weight loss per unit area (g cm^{-2})--This is the difference between the weight of the flight and control samples before and after the mission, ratioed to the exposed area of the flight samples.

- The sample thickness loss (cm)--This is obtained from the difference in thickness of the flight samples before and after the mission. Depending on the magnitude of the difference, one can determine the degree of coating loss attributable to the space environment.

- A percent thickness loss--This is based on the same assumptions used for the measurements of the coating thickness.

- A comparison of the spectral reflectance and the integrated absorption and emittance of the coatings before and after space environment exposure--The measurements were made using the P.E. λ-9 spectrophotometer.

- Surface analysis of the samples--The ESCA probe was used to provide elemental/ chemical composition of the samples within a depth of 100 Å (up to some 50 monolayers) employing x rays to emit electrons.

- Photographic and microscopic documentation--This shows the reference and flight sample surface appearance and related evaluation of the changes that may have occurred following space exposure.

4. SAMPLE ANALYSES

1000 Å of SiO_x on VDA Kapton

Visual Inspection--The surface was highly reflective with some raised areas. The flight sample surface was slightly duller than the reference sample, with extremely fine discontinuities over the entire surface.

High-Magnification Inspection--The shape of the discontinuities was not discernible at 200 X magnification. There were no pinholes in the vacuum-deposited Al.

ESCA Analysis--The composition of both the flight and reference samples were similar, as shown by the spectrogram in Figure 6. The concentrations of Si and O remain constant through the thicknesses of both samples. Erosion may have been uniform over the surface. Peaks of silicone and oxygen are found within the various thicknesses.

Radiative Analysis--The reflectances vs. wavelengths are shown in Figure 7. The exposed sample shows an improved reflectance below 450 nm and above 700 nm. The integrated properties are $\alpha = 0.127$ and $\varepsilon = 0.023$ for the flight sample, and $\alpha = 0.155$ and $\varepsilon = 0.025$ for the reference sample.

Physical Analysis--The mass loss of the flight sample was 3.3×10^{-5} g or about 8.9×10^{-6} g cm^{-2} of the exposed area. The thickness change amounted to 3.032×10^{-3} cm, corresponding to about 0.994% of the total sample thickness.

Oxygen Erosion--The change in thickness, 3.032×10^{-3} cm, is considerably more than the SiO_2 thickness of 1000 Å (1×10^{-5} cm). Some of the VDA Kapton was eroded. One cannot establish a reaction rate constant because the measured mass loss and thickness may include changes due to the sample's outgassing losses.

100Å In_2O_3 on VDA/Kapton

Visual Inspection--Figures 8 and 9 reveal uniformly oriented serrations in the highly reflective, gold-colored Kapton surface. Brushed marks on the aluminized surface are opaque with a golden hue and an aluminized color visible on only a few small areas.

High-Magnification Inspection--An etched, frosted appearance is visible at the brush marks under the undamaged areas.

ESCA Analysis--The flown coating sample (Figure 10) has a rough surface with visible erosion and delamination. The non-exposed surface appears shiny at the outer edge, with no visible damage. The exposed surface is severely eroded and gray in color, with scratch

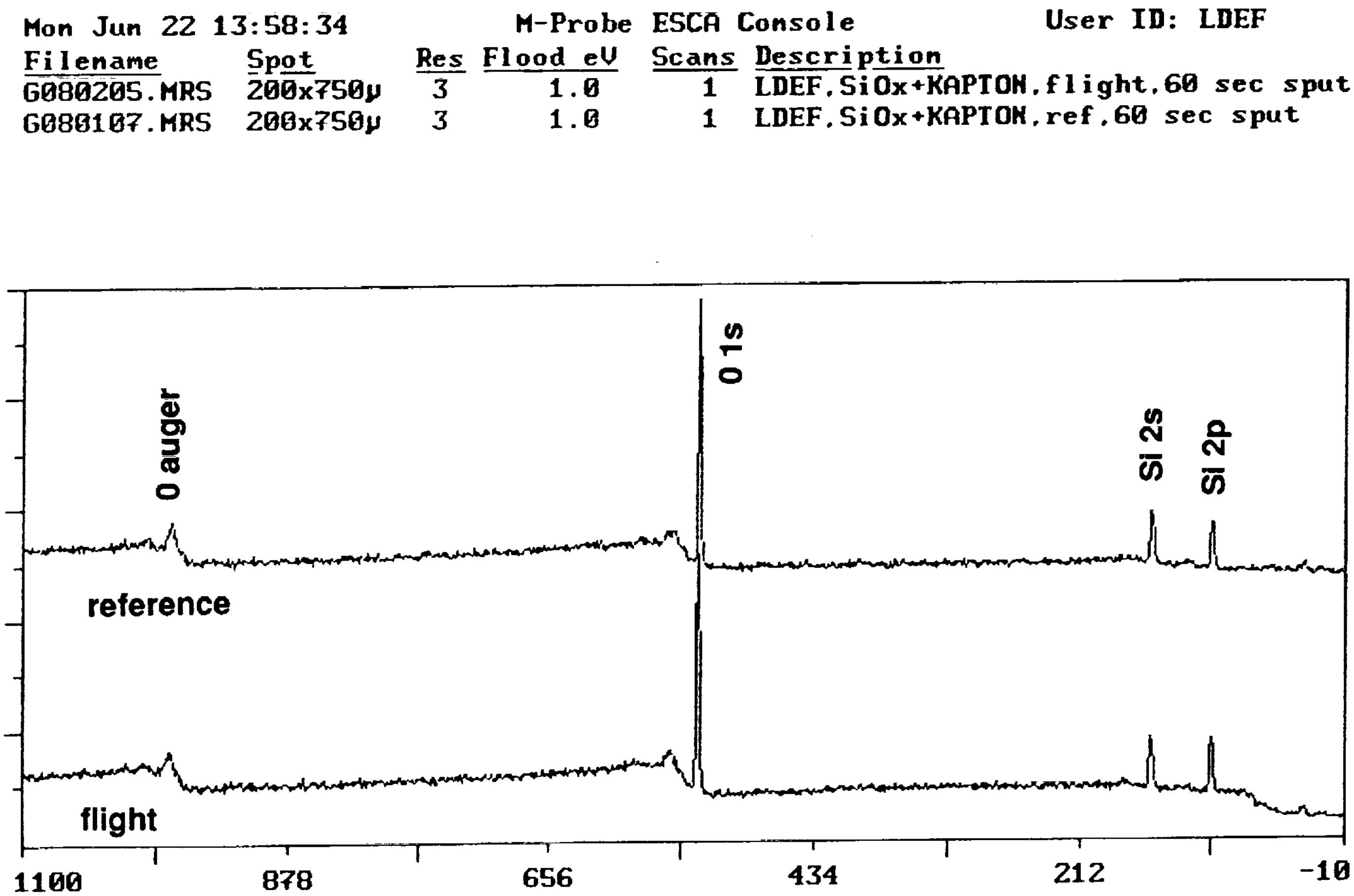

Figure 6. ESCA spectrum of 1000 Å of SiO_x on VDA/Kapton.

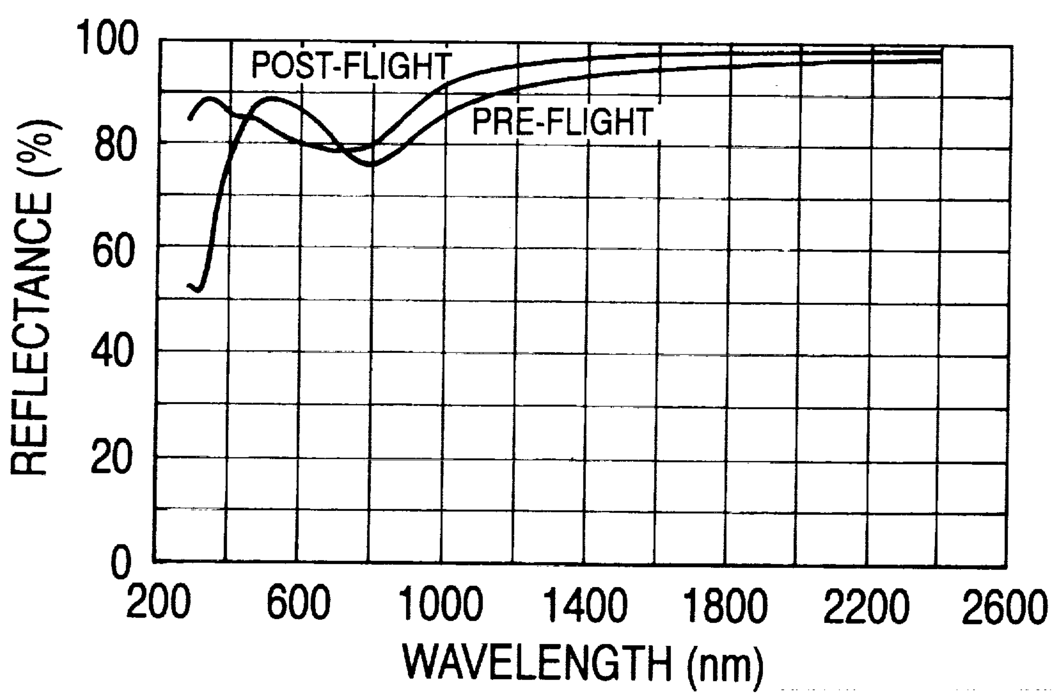

Figure 7. Reflectance of 1000 Å SiO_x coating on VDA/Kapton sample.

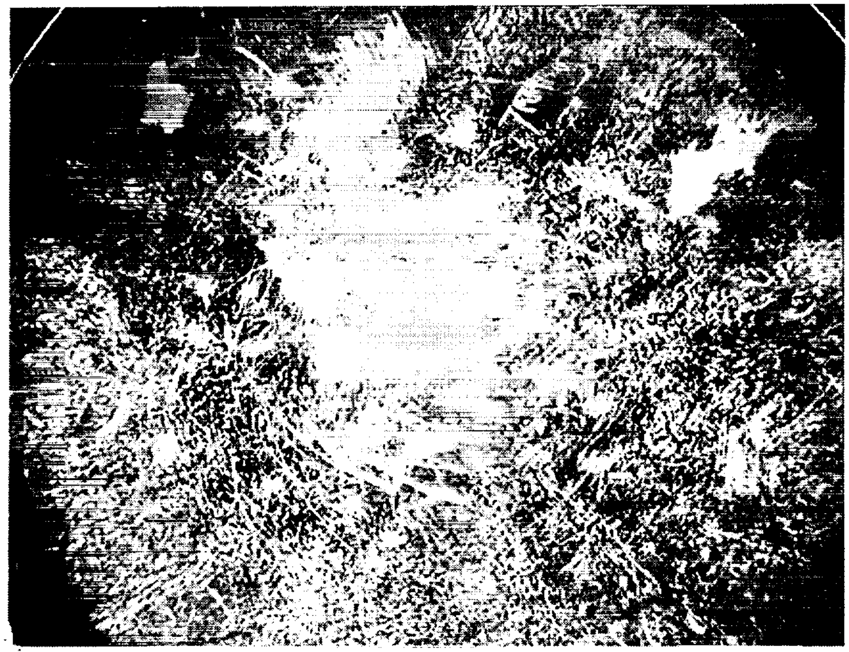

Figure 8. 100 Å In_2O_3 on VDA/Kapton (5.5 X).

Figure 9. VDA/Kapton/InO_x (5.5 X).

Mon Jun 22 13:50:46 M-Probe ESCA Console User ID: LDEF

Filename	Spot	Res	Flood eV	Scans	Description
G072910.MRS	200x750µ	3	1.0	1	LDEF.VDA#2-KAPTON.center.60sec sput
G072903.MRS	200x750µ	3	1.0	1	LDEF.VDA#2-KAPTON.reference. 60 sec

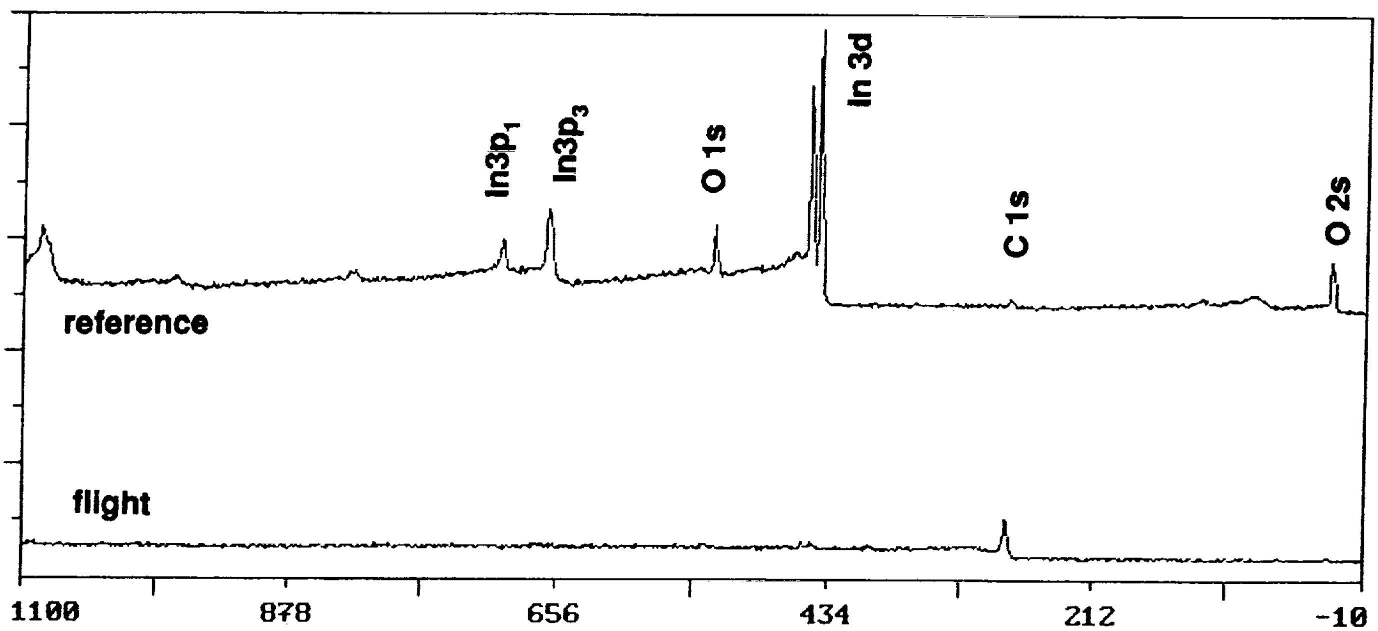

Figure 10. ESCA spectrum of sample with 100 Å of In_2O_3 on VDA/Kapton.

marks around the eroded area. The scratched and eroded areas are made up mostly of Kapton, with some traces of indium. The indium at those locations measured up to 0.95 atomic % while at the unexposed surfaces, the indium was 7 atomic %.

Radiative Analysis--Figure 11 shows the reflectances of the exposed and reference samples. About 10% reflectance loss occurred at wavelengths below 450 nm and about 5% loss occurred between 600 and 1600 nm. The integrated values are 0.391 absorption and 0.547 emittance for the flown sample and are 0.363 and 0.564, respectively, for the reference sample.

Physical Analysis--The mass loss for the sample was 0.001867 g, or about 5.37×10^{-4} g cm^{-2} of exposed area. The thickness change amounted to about 5.08×10^{-3} cm, corresponding to about 1.538% of the total thickness.

Oxygen Erosion--The 100 Å (10^{-6} cm) of In_2O_3 and a considerable amount of the VDA/ Kapton were eroded. In addition, considerable material and thickness must have been lost by outgassing in space. Not knowing if bakeout in vacuum was performed on the material before launch, it is not possible to estimate the reaction efficiency of the indium. However, the various analyses have indicated that the indium was completely eroded. The reaction rate for the Kapton is known to be about 3×10^{-24} cm^3/atom from other orbital tests.

Devolatized RTV-615 Bonded on Al with SS 4155 Primer

Visual Inspection--The surface is clear and transparent with no noticeably changed features (Figures 12 and 13).

High-Magnification Inspection--Optical magnification shows banded networks with areas of contamination (possibly impacts) at focal points of several bands (Figures 12 and 13). The network of crack lines may have orignated from solar exposure and from additional material losses causing thermal cracking.

ESCA Analysis--The erosion pattern is similar to that of the sample consisting of the same RTV with K_2SiO_3 and ZnO pigment. The flight sample shows carbon content of 1.5 atomic %, while the reference sample has 35 atomic %. The Si concentration did not change, while the O concentration doubled in the flight sample (Figure 14).

Radiative Analysis--The flight sample experienced a loss of about 5% in reflectance throughout the measured range of wavelength with respect to that of the reflectance sample. The integrated properties are: $\alpha = 0.489$ and $\varepsilon = 0.819$ for the flight sample, and $\alpha = 0.432$ and $\varepsilon = 0.824$ for the reference sample (Figure 15).

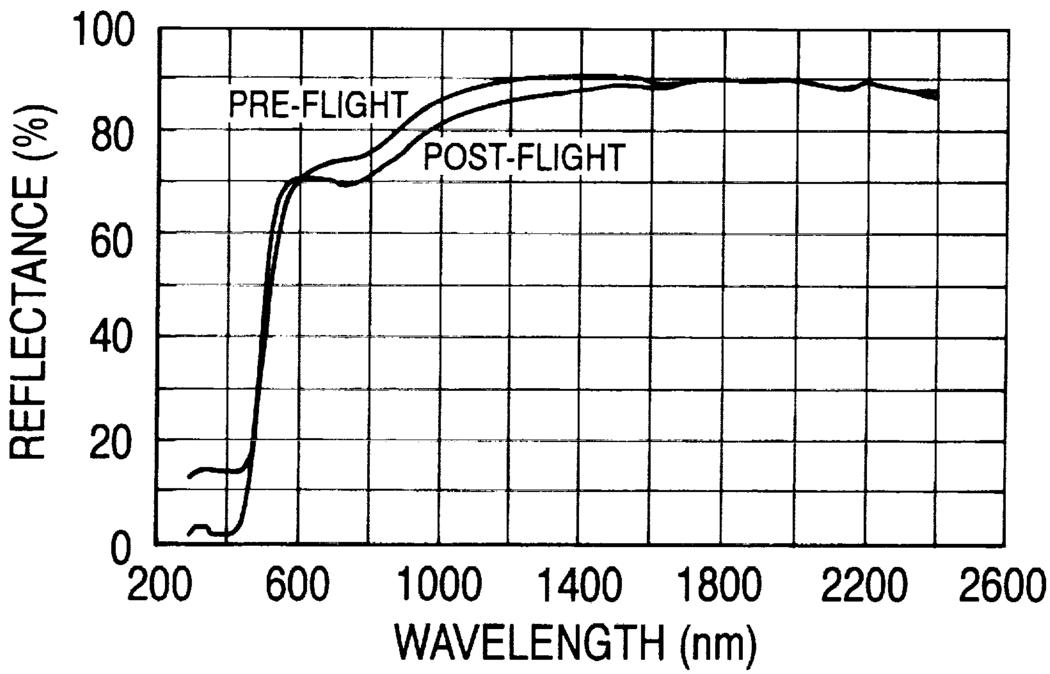

Figure 11. Reflectance of 100 Å InO_3 coating on VDA/Kapton sample.

Figure 12. Devolatized RTV-615 bonded on Al with SS 4155 Primer (200 X).

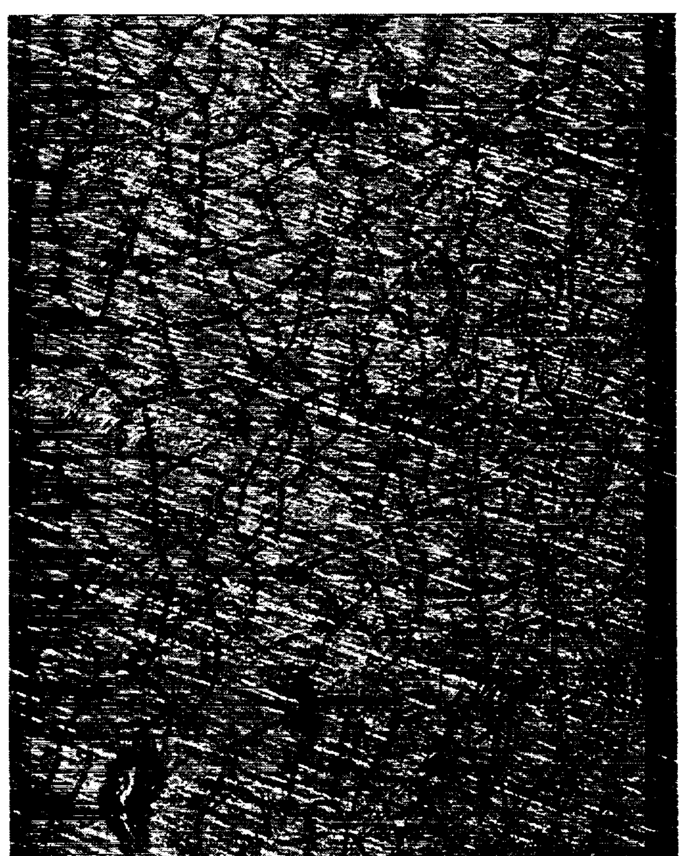

Figure 13. Devolatized RTV-615 bonded on Al with SS 4155 Primer (25 X).

Mon Jun 22 14:22:28 M-Probe ESCA Console User ID: LDEF

Filename	Spot	Res	Flood eV	Scans	Description
G091305.MRS	400x1000µ	3	1.0	3	LDEF,RTV-clear,flight,60 sec sput
G091205.MRS	400x1000µ	3	1.0	3	LDEF,RTV-clear,reference,1 min sput,

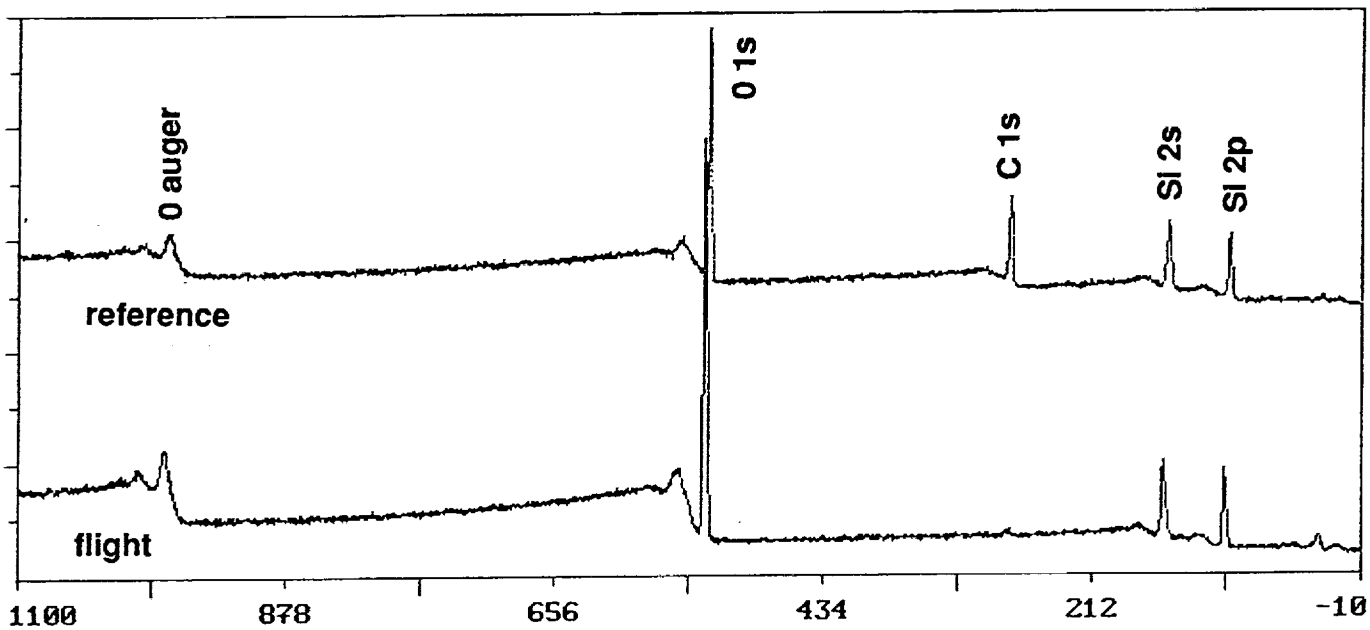

Figure 14. ESCA spectrum of sample with RTV-615 bonded on Al with SS 4155 primer.

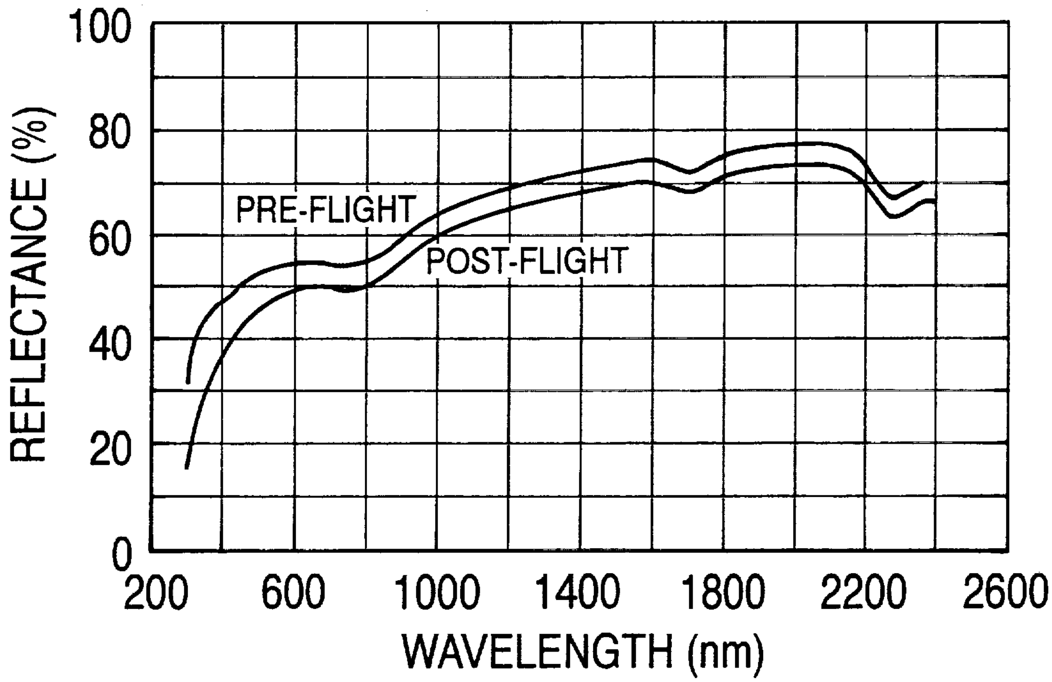

Figure 15. Reflectance of RTV-615 clear coating on aluminum.

Physical Analysis--The mass loss was 0.0037 g, or about 8.983 x 10^{-3} g cm^{-2} of the exposed area. The thickness change amounted to about 8.63 x 10^{-3} cm, corresponding to about 2.617% of the total thickness.

Oxygen Erosion--The change in thickness, 0.0034 in. (8.63 x 10^{-3} cm), is considerably less than the thickness of the RTV and primer 0.0167 in. (4.24 x 10^{-2} cm). Under the assumptions that the loss was the result of the oxygen erosion, we could calculate the reaction efficiency.

However, calculations to estimate the reaction efficiency using the above data indicate a considerable oxygen erosion, much larger than the value of 6.25 x 10^{-26} cm^3/atom reported in reference 3. The discrepancy in order of magnitude must be assumed to have been produced by loss of material from outgassing.

RTV-615/Silicate-Treated ZnO

Visual Inspection--The flight sample surface appears slightly glossy and white, with raised agglomerated particles originating from the glossy matrix surface. The reference sample is flat white.

High-Magnification Inspection--The sample surface is shown in Figures 16 and 17.

ESCA Analysis--This analysis (Figure 18) indicated considerable difference in the concentrations of carbon (C) between the flight and the reference specimens. The concentrations of Si between the two are about equal and constant through the thicknesses. The O and C concentrations differ. The C concentration decreases by 21 atomic % and O increases by 18 atomic % after 1 minute of etching. On the other hand, for the reference sample, the C decreases by 7 atomic % concentration and the O increases by 2 atomic % for the same etching time. No Zn or K peaks were found, even though the silicone was filled with potassium silicate and ZnO, indicating that they had eroded or that they had penetrated into deeper layers.

Radiative Analysis--The reflectance versus wavelength is shown in Figure 19. It shows some loss between 400 and 700 nm and between 1800 and 2100 nm. The integrated absorption is 0.201 and the emittance is 0.891 for the flown sample and 0.190 and 0.907, respectively, for the reference sample.

Physical Analysis--The mass loss was 8.27 x 10^{-4} g, or about 2.33 x 10^{-4} g cm^{-2} of exposed surface. The thickness change amounted to 3 x 10^{-3} in. (7.78 x 10^{-3} cm), corresponding to about 2.142% of the total thickness.

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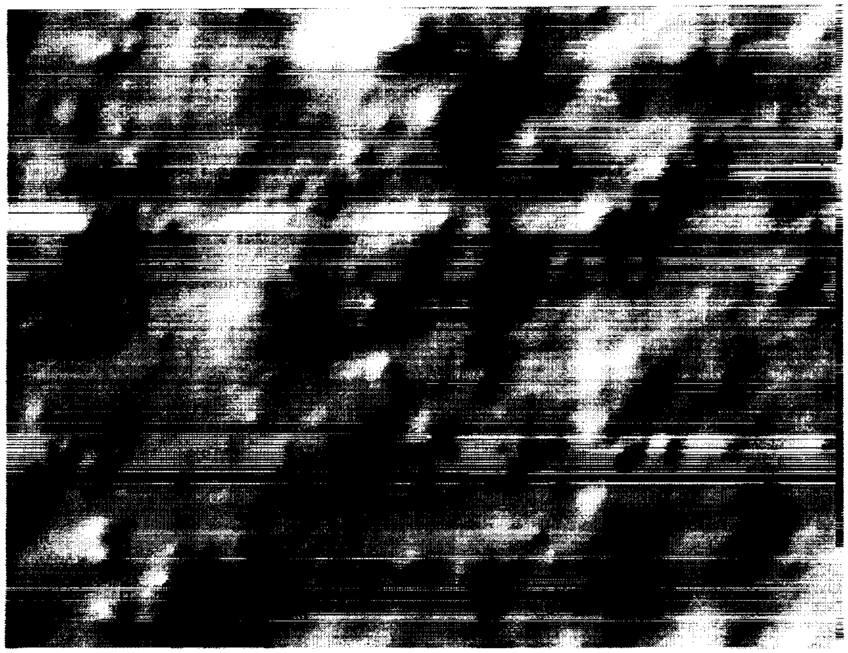

Figure 16. RTV-615/Silicone-Treated ZnO (38.5 X).

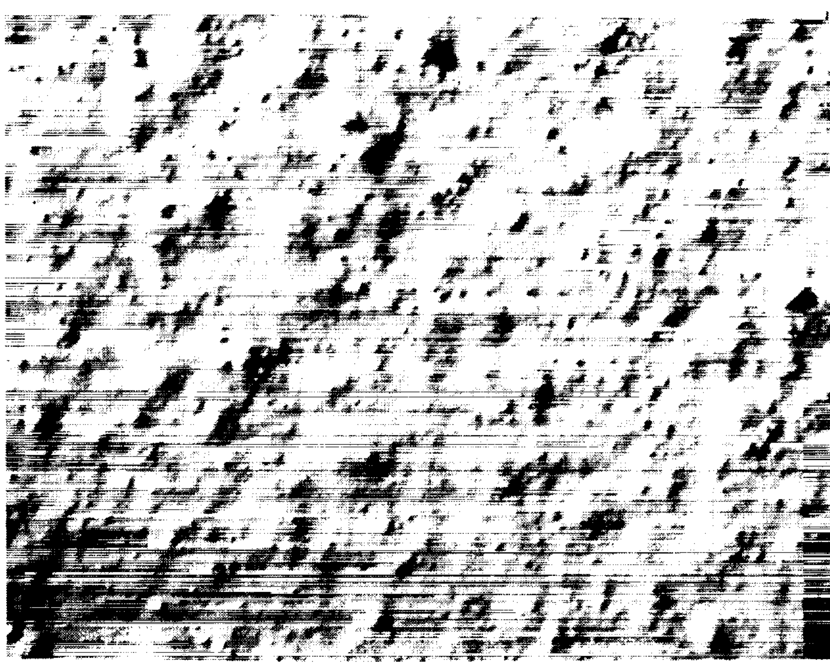

Figure 17. RTV-615/Silicone-Treated ZnO (16.5 X).

Mon Jun 22 14:20:25 M-Probe ESCA Console User ID: LDEF

Filename	Spot	Res	Flood eV	Scans	Description
G091107.MRS	400x1000µ	3	1.0	3	LDEF,RTV-white,flight,3 min sput
G090807.MRS	400x1000µ	3	1.0	3	LDEF,RTV-white,ref,3 min sput,new lo

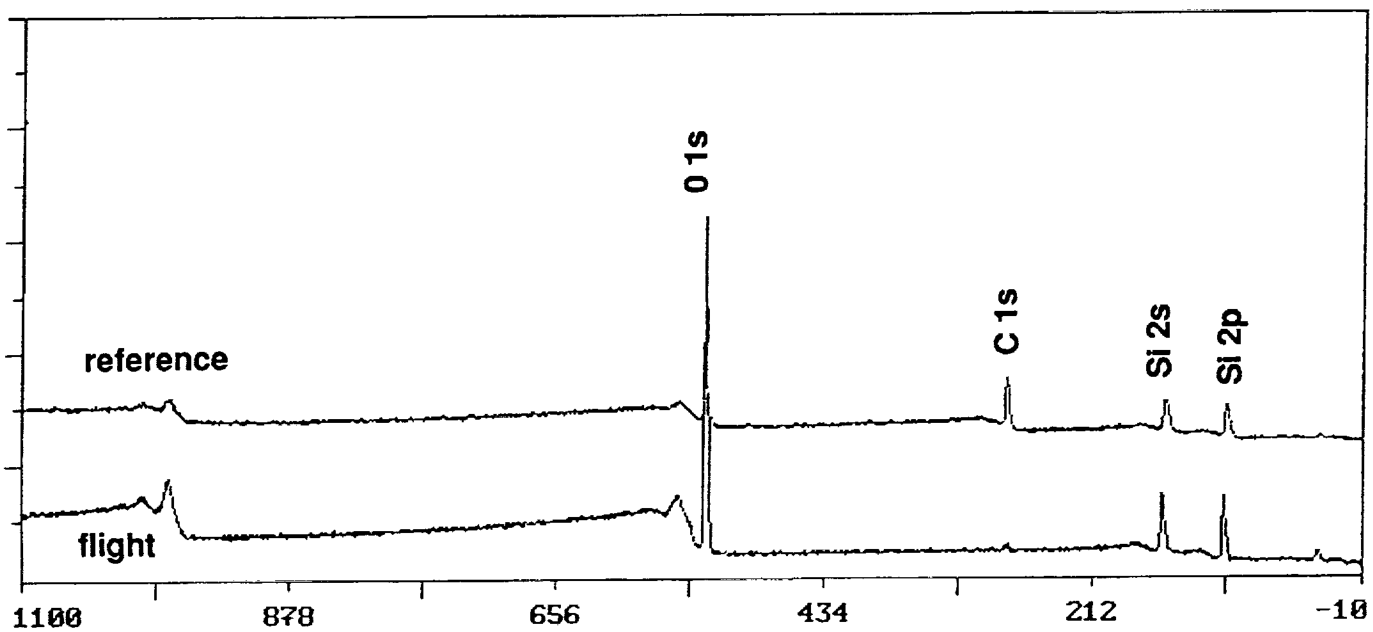

Figure 18. ESCA spectrum of Sample with RTV-615 Silicone Treated with ZnO Bonded on Al.

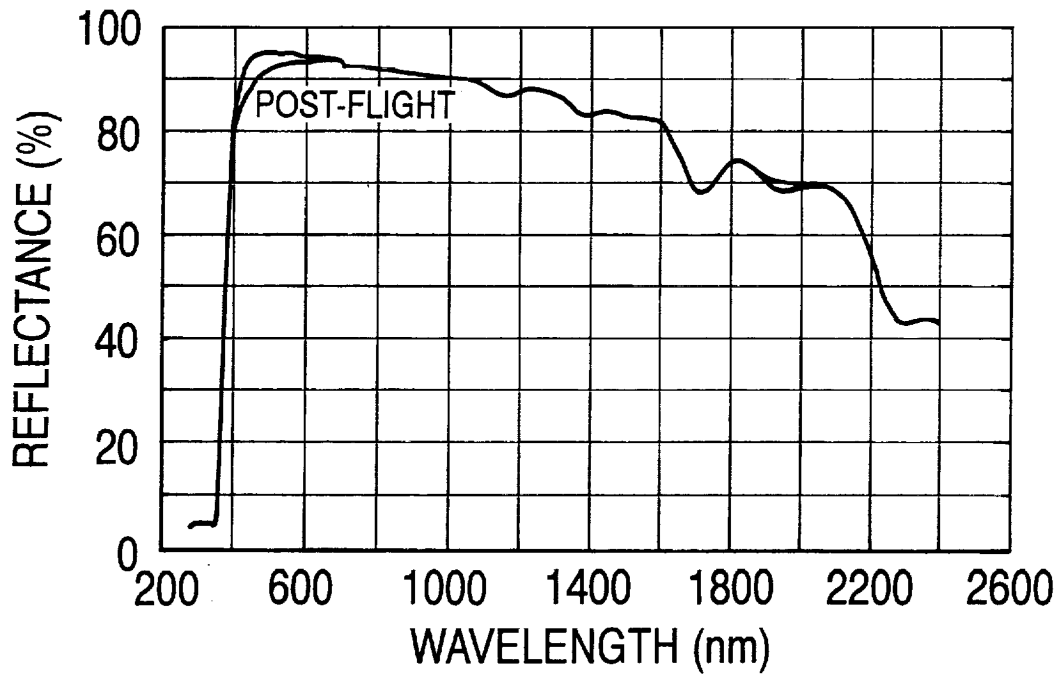

Figure 19. Reflectance of RTV-615/Silicone-Treated ZnO.

Oxygen Erosion--Both the RTV and the silicate were eroded. The actual erosion and mass thickness are not known because of the possible loss by outgassing, and the calculation for the reaction efficiency could be erroneous. But, as indicated, the erosion did occur.

5. CONCLUSIONS

The four material coatings aboard the LDEF that were exposed directly to the space environment at an orbit of 260 nautical miles for 10 months, beginning 1 month after launch, have exhibited the following.

- **1000 Å of SiO_x on Kapton**--The sample of SiO_x was uniformly eroded. The concentrations of O and Si remained constant. Some improved reflectance occurred below 450 nm and above 700 nm.

- **100 Å In_2O_3 on VDA/Kapton**--The sample was severely eroded, with the indium reduced to less than 0.95 atomic % in comparison to the unexposed sample at 7 atomic %. The color changed from gold to gray. Kapton was exposed to the eroded areas and it exhibited substantial erosion. Losses of 5% to 10% in reflectance resulted below 450 nm and between 600 and 1600 nm.

- **RTV-615 Devolatized on Aluminum**--The sample surface shows erosion and banded networks originating from focal points. Carbon content dropped significantly. The Si concentration remained constant while the O concentration doubled with respect to the reference sample. Reflectance losses of about 5% occurred throughout the analyzed spectrum.

- **RTV-615 Silicate-Treated ZnO**--The flight sample surface changed from flat white to slightly glossy white. The C concentration decreased by about 21 atomic % while the O concentration increased by 18 atomic %, as it etched into the surface. A comparable etching of the reference sample indicates a C drop of 7 atomic % and an O increase of 2 atomic %. No Zn and K were found, indicating either erosion or penetration deeper into the coating. Some loss of reflectance is noted at the wavelengths between 400 and 700 nm, and between 1800 and 2100 nm. The silicone was eroded.

An evaluation of the O reaction efficiency for the coatings exposed to a total O fluence of about 2.6×10^{20} atoms cm^{-2} was not possible. The material losses and recessions, quite certainly, included outgassing products, but these could not be determined. However, from the above analyses, general indications of these coatings' performance in orbit was possible.

6. ACKNOWLEDGMENTS

The authors are grateful to the following coworkers for their contributions to this work: Gloria Oh, for the ESCA analyses; Wanda Peters, for the reflectance measurements; and Diane Kolos, for the microscopic examination of the samples.

7. REFERENCES

1. Young, Philip R. and W. S. Slemp, *Analysis of the LDEF-Exposed Silvered FEP Teflon Thermal Blanket Material*, NASA TM 104096, December 1991.

2. Kinard, William H. and G. D. Martin, *Long Duration Exposure Facility Space Environment*, NASA CP 3134, p. 34, 1991.

3. Banks, B., et al., *Atomic Oxygen Effects on Materials*, NASA CP 3035, June 1988.

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13. ABSTRACT *(Maximum 200 words)*
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