N93-29694

EFFECTS OF LONG TERM SPACE ENVIRONMENT EXPOSURE ON OPTICAL SUBSTRATES AND COATINGS (S0050-2)

Keith Havey, Arthur Mustico, and John Vallimont
Eastman Kodak Company
Rochester N.Y.

Eastman Kodak Company included twelve substrate and coating samples on the LDEF structure. There were three Fused Silica and three Ultra Low Expansion (ULE™) uncoated glass samples, two ULE™ samples with a high reflectance silver coating, two Fused Silica samples with an antireflectance coating, and two Fused silica samples with a solar rejection coating. A set of duplicate control samples was also manufactured and stored in a controlled environment for comparison purposes.

Kodak's samples were included as a subset of the Georgia Institute of Technology tray, which was located on row 5-E, tray S0050-2. This placed the samples on the trailing edge of the structure, which protected them from the effects of atomic oxygen bombardment.

An evaluation of the flight samples for effects from the 5 year mission showed that a contaminant was deposited on the samples, a micrometeoroid impact occurred on one of the samples, and the radiation darkening which was expected for the glass did not occur. The results are listed below in more detail.

SAMPLE DESCRIPTION

Twelve samples were chosen for inclusion in the radiation experiment. The sample size was 1.250 inches in diameter and .040 inches thick, consistent with an ANSI break strength size. Both the faces and edges of the samples were polished. The substrate materials and coatings selected for the samples were chosen from those which have been specified on previous or current space flight optics.

The orientation of the coating samples in the LDEF simulated as closely as possible their actual mounting configurations when used on prime hardware. The high reflectance coating was on the outside of the sample facing the environment (the inside surface was also coated). The solar reflectance coating also faced the environment, but was on the inside face of the sample, and was protected by its fused silica substrate. The antireflectance coating was on both sides of the substrate.

All of the samples were measured for transmission, reflection, and stress prior to launch and after retrieval. Wash and tape tests were also performed on the samples to insure coating durability and adhesion. The set of control samples was also tested for comparison purposes.

The samples were delivered directly to NASA and assembled into the LDEF tray in a clean room environment to insure the cleanliness of the samples was maintained. A fine vacuuming of the samples was performed and close up pictures were taken just prior to sealing of the LDEF tray. The method of securing the samples in the tray is shown in Figure 1. A picture of the flight samples mounted in the LDEF tray is shown in Figure 2.

Twelve baseline samples identical to the space radiation samples were also simultaneously manufactured and tested. The purpose of these samples was to provide a direct comparison between exposed and non-exposed samples. The baseline samples were double sealed in nylon and anti-static polyethylene film and stored until retrieval of the flight samples.

POST FLIGHT EVALUATION

A thorough evaluation was performed on the LDEF samples for effects from the space environment. This included contaminant analysis, measurement of optical performance, induced stress, and BRDF. The control samples were also measured and the results compared to the flight data. Specific details of this evaluation follow.

MICROMETEOROID IMPACT

A micrometeoroid impact site was found on one of the samples. The impact crater measured .3 mm in diameter by .03 mm deep. Multiple fractures occurred in the glass at the impact site and are shown in Figure 3.

SAMPLE CLEANING

One sample of each type was cleaned after the initial optical performance measurements were made. The contaminate, a light brown in coloring, was removed fairly easily from all of the samples using normal cleaning methods and Isopropyl alcohol, with the exception of the Antireflection coated sample. On this sample, the contaminate could not be removed. It was not affected by either the alcohol, Toluene, Methyl Ethyl Ketone, Cyclohexene, 50/50 Nitric-Sulfuric acid, or heated Tri-Chlorobenzene. Only after exposure to an Oxygen plasma did some reduction in coloring occur. Three hours exposure to an Oxygen plasma reduced the brown discoloration, and increased the spectral transmission through the sample, as can be seen in Figure 4. The sample will be given additional exposure to the Oxygen plasma and its transmission will be measured after each exposure.

RADIATION DARKENING

Some radiation darkening could be expected of the Ultra Low Expansion (ULE™) glass which is not a radiation tolerant glass. No darkening of either the ULE™ or the Fused Silica glass was evident. There was no change in the transmission values of the pre-flight and after cleaning measurements, as shown in Figures 5 & 6.

OPTICAL PERFORMANCE

The flight samples were measured for optical performance from 350 to 1200 nm. Figures 4 thru 10 document the performance pre-flight, after flight, and after cleaning. As the figures show, all of the substrates and coatings experienced a significant performance reduction after flight, but after cleaning (except for the Antireflection coated sample which we couldn't clean), their performance returned to the pre-flight measured values. Of interest is the fact that the density of the contaminant deposited on the samples varied between coatings and substrate material. As an example, the transmission of the uncoated Fused Silica sample was reduced from 94% to 68% at 350 nm, while that of the uncoated ULE sample was reduced from 94% to 45%.

BRDF MEASUREMENT

A high reflectance silver coated flight sample (with contaminant) and a control sample were measured for Bidirectional Reflection Distribution Function. An increase in scattered light was measured on the flight sample versus that of the control sample, as shown in Figure 11.

STRESS MEASUREMENT

The samples were measured photoelastically for stress before and after flight using birefringence polarimetry. The control samples were also measured for comparison purposes. Table 1 lists the measured substrate stress values, the results of which are summarized as follows:

1) On the uncoated glass and high reflectance silver coated samples, the contaminant, on the average, induced a compressive stress of 42 psi. After cleaning, the stress levels closely matched those of the pre-flight measurements.

2) On the fused silica anti-reflectance coated sample, the contaminant did not induce any measurable stress.

3) A stress change could not be measured on the solar rejection coated samples due to the high level of stress in the coating, and the variation in stress between samples. A reduction in the stress levels in this coating was measured on both the flight and control samples.

4) No significant stress change was measured between the flight samples after cleaning and the control samples.

CONTAMINANT ANALYSIS

The contaminant on the samples was analyzed using X-ray Photoelectron Spectroscopy (XPS), which showed it to be a thin layer of polymer which contained silicon. The contaminant on the Antireflection coated sample and uncoated ULE™ sample appeared to be slightly different than that deposited on the other samples. On these two samples the energy peaks from the silicon, as listed in table 2, were representative of the binding energy of silicone rubber.

On the other samples, the energy peaks were higher, and more representative of the binding energy of SiO2. However, neither the relative atomic percentages or the relative sizes of the silicon and oxygen peaks from the XPS conclusively prove that the contaminant is a residue from the mounting rubber gasket.

Depth sputtering through the Antireflection coating, as can be seen in Figure 12, clearly shows the SiO2/TiO2 layers for both the control and flight samples. However the flight sample has a layer at the surface approximately 30 Angstroms thick, rich in carbon and silicon. The carbon content rises as the contamination layer is sputtered away and disappears at the contamination - antireflection layer interface. The total time required to sputter to the bottom of the SiO2/TiO2 layers was almost identical for both the flight and control samples. Since the hardness of the coating on both samples was similar, the sputtering rates would have been equivalent, making the total coating thicknesses the same. This indicates that either the top surface of the flight coating has been removed and replaced with the silicon/carbon layer, or the silicon/carbon has fused into the SiO2 layer.

SUMMARY

The 5+ year exposure of the samples to the space environment resulted in a contaminant being deposited on the samples which reduced optical performance, induced stress, and varied in composition between the samples depending on the coating. One sample received a micrometeoroid impact. After cleaning the contaminant from the samples, all of the coatings and substrates, with exception of the anti-reflection coated samples, returned to their original pre-flight performance.

LDEF SAMPLE STRESS DATA				
	STRESS PSI			
SAMPLE DESCRIPTION	PRE-FLIGHT	POST-FLIGHT	AFTER CLEANING	CONTROL SAMPLE
FUSED SILICA UNCOATED	9.26 T (6)	35.3 C (1)	2.27 T (1)	10.7 T (3)
ULE™ UNCOATED	9.51 T (6)	38.5 C (1)	8.0 T (1)	17.1 T (3)
ULE™ H. R. COATED (both sides)	14.5 T (6)	33.1 C (1)	9.0 T (1)	15.4 T (3)
FUSED SILICA A. R. COATED	15.6 T (6)	16.4 T (2)	-------	20.1 T (3)
FUSED SILICA S. R. COATED	676 C (6)	553 C (1)	569 C (1)	557 C (3)
FUSED SILICA S. R. COATED (uncoated side)	210 T (6)	185 T (1)	160 T (1)	150 T (3)
	( ) NUMBER OF SAMPLES MEASURED "T" INDICATES TENSION "C" INDICATES COMPRESSION			

Table 1. Stress measurements in LDEF samples

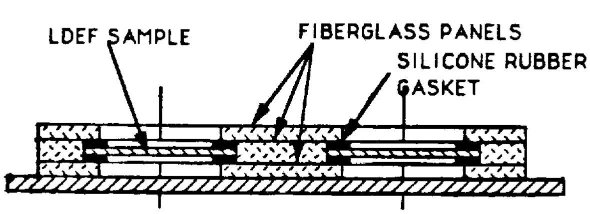

Figure 1. Sample mounting

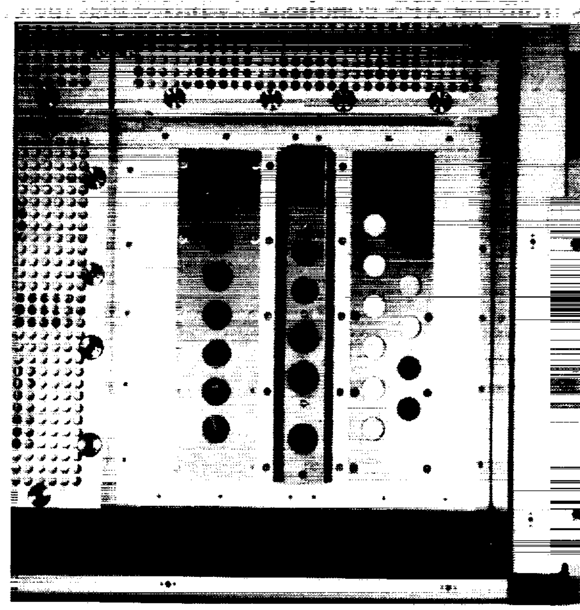

Figure 2. Samples mounted in LDEF tray (12 samples on right)

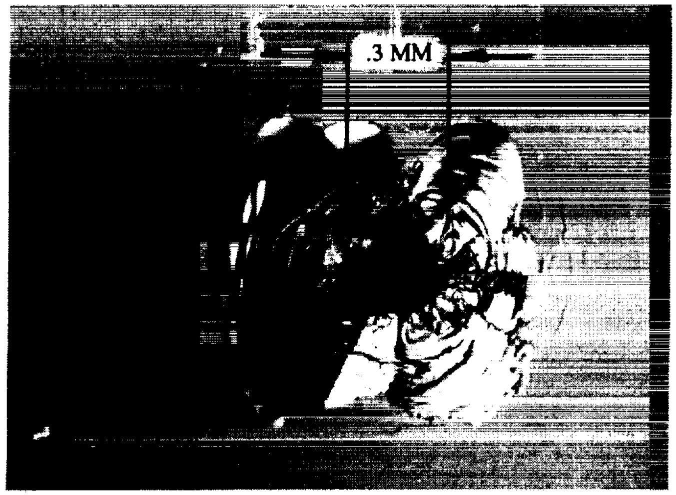

Figure 3. Micrometeoroid impact crater

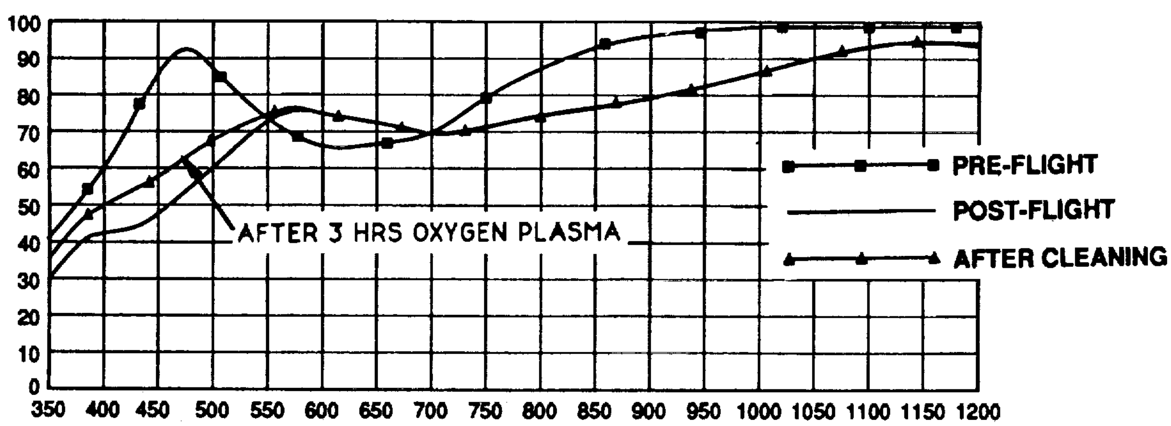

Figure 4. Spectral transmission of fused silica anti-reflection coated at 1.06λ

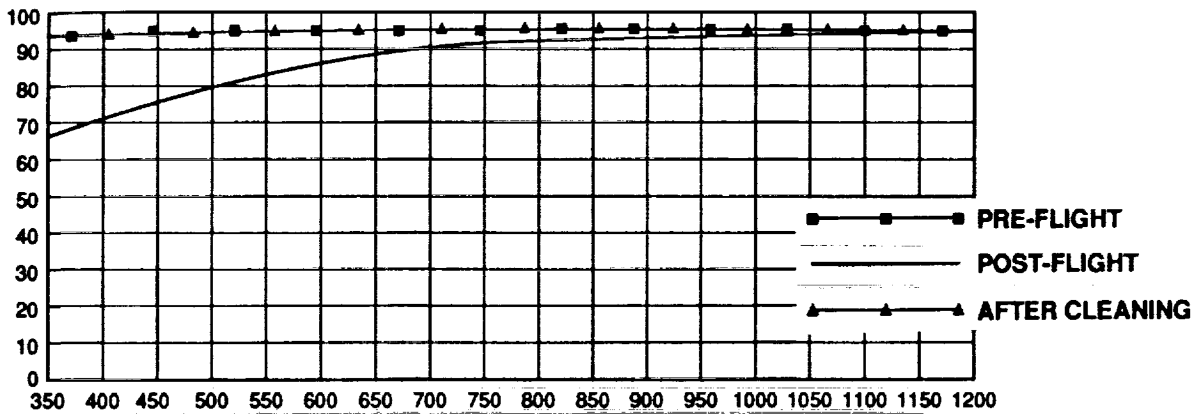

Figure 5. Spectral transmission of uncoated fused silica

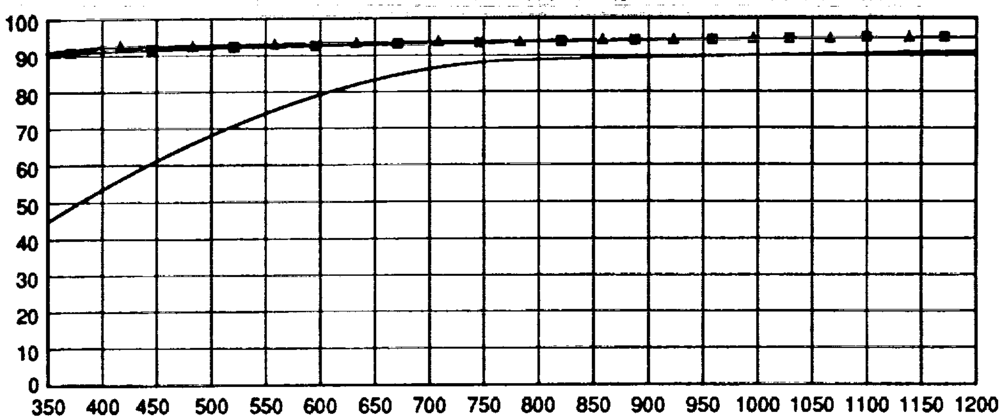

Figure 6. Spectral transmission of uncoated ULE™ sample

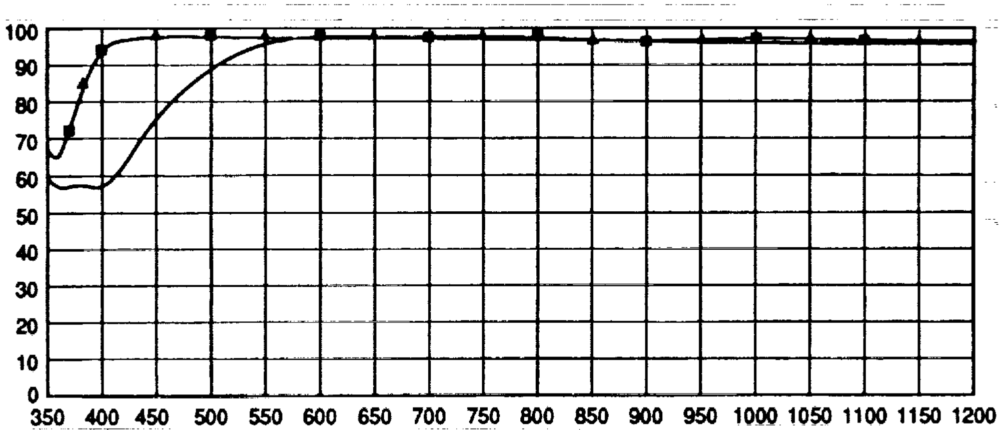

Figure 7. Spectral reflection of ULE™ H.R. silver coated outboard

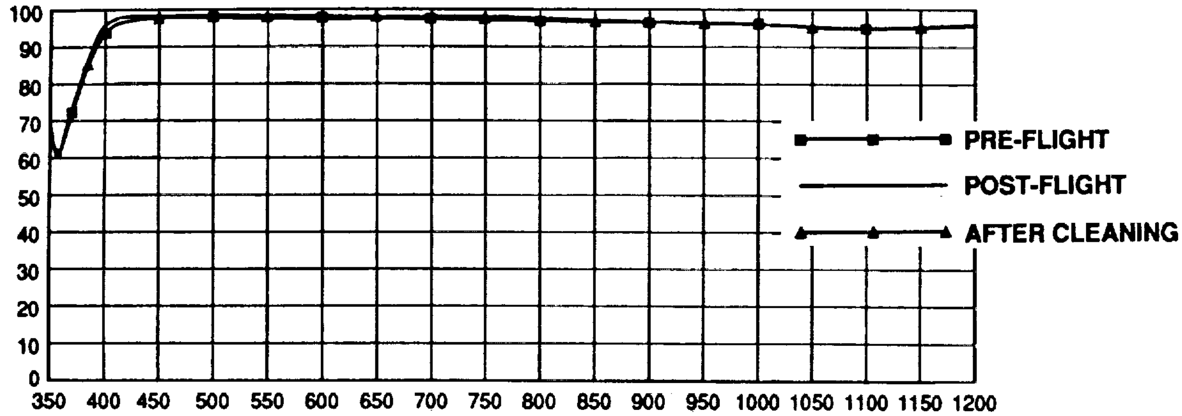

Figure 8. Spectral reflection of ULE™ H.R. silver coated inboard

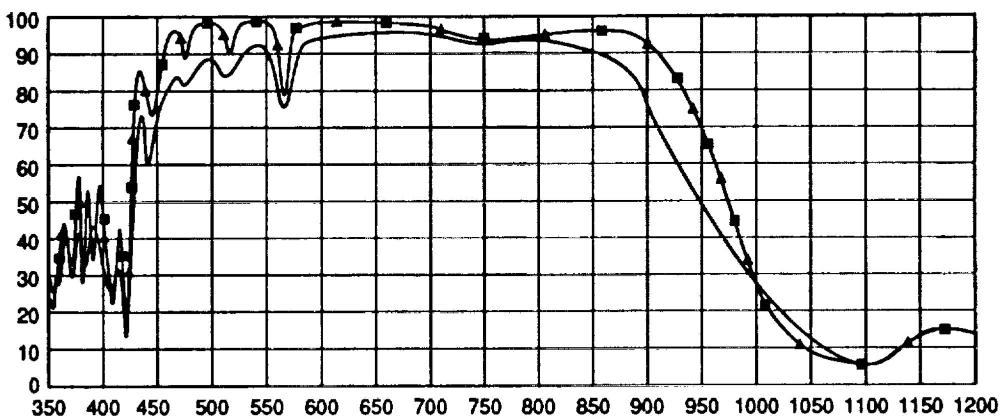

Figure 9. Spectral reflection of fused silica solar rejection coated

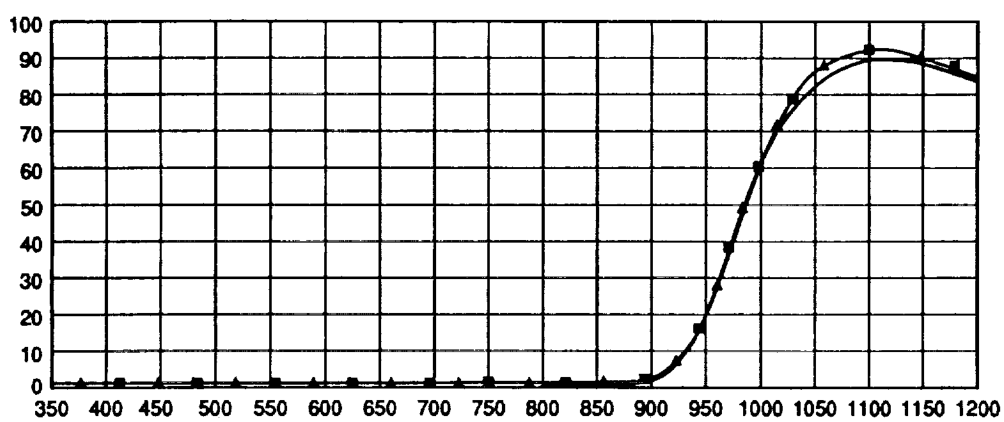

Figure 10. Spectral transmission of fused silica solar rejection coated

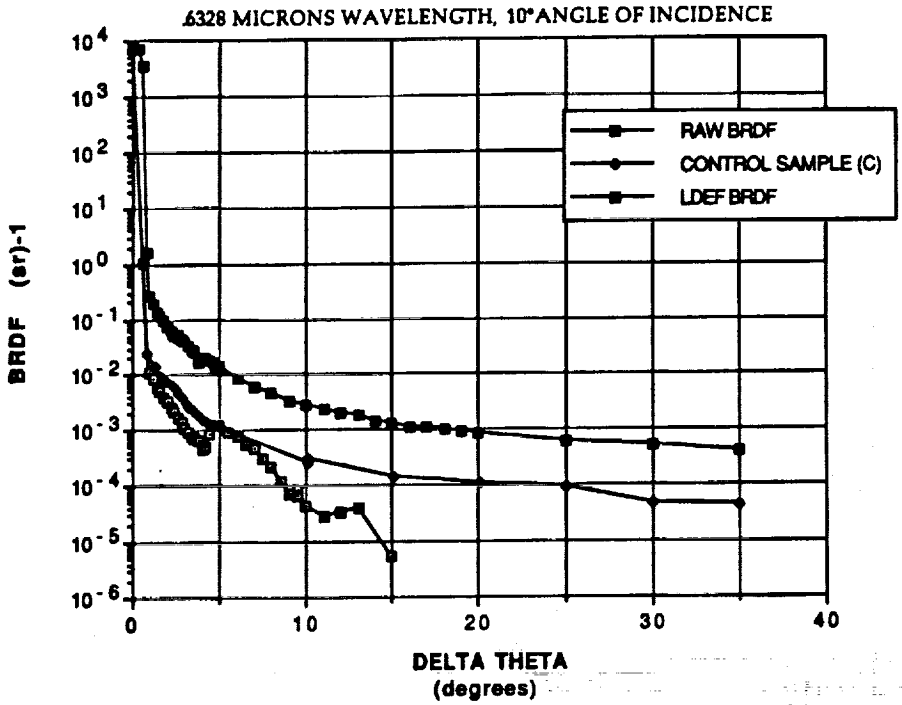

Figure 11. BRDF measurement of LDEF flight and control sample high reflectance silver coated

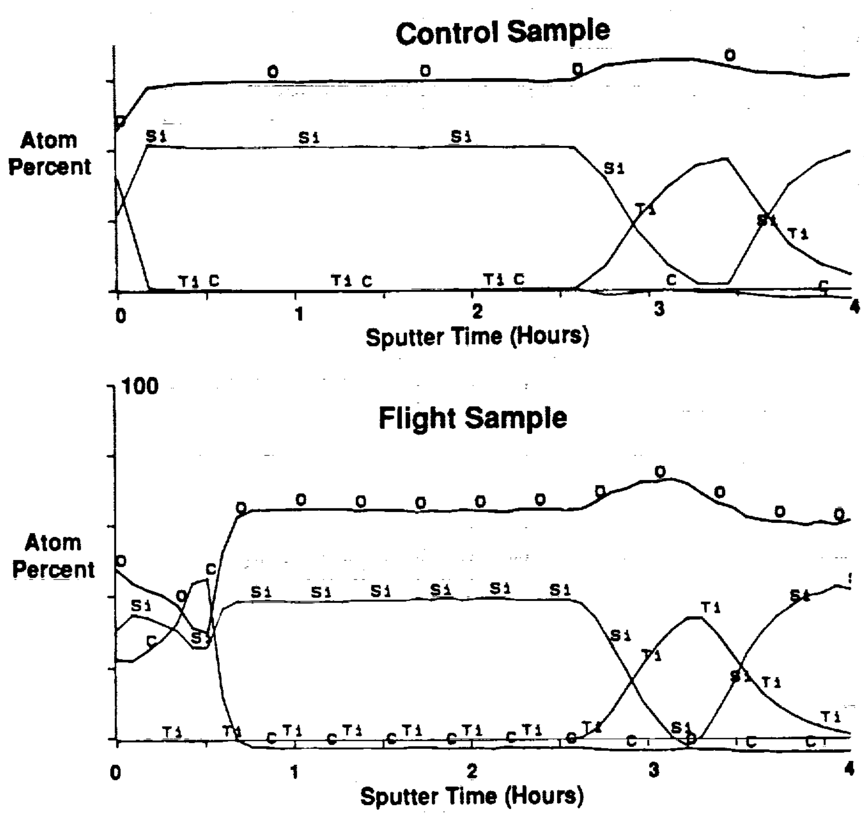

Figure 12. XPS profiles antireflection coated fused silica

LDEF Witness Samples Atomic Percents, Ratios and Peak Positions Carbon, Oxygen and Silicon						
		O		Si		
	C	Position (eV)	Area (%)	Position (eV)	Area (%)	O/Si Ratio
Fused Silica Uncoated Control Sample	25.5	532.2	45.2	103.0	24.2	1.87
Fused Silica Uncoated Flight Sample	32.2	532.7	41.7	103.2	25.0	1.67
Ultra Low Expansion Uncoated Control	25.0	532.1	45.8	102.9	24.1	1.90
Ultra Low Expansion Uncoated Flight	53.0	532.2	24.1	102.4	22.6	1.07
High Reflectance ULE™ Control Sample	62.5	531.7	30.6	na	na	na
High Reflectance ULE™ Flight Sample	31.7	532.7	43.1	103.1	23.5	1.83
Anti Reflectance Fused Silica Control	28.9	532.7	45.6	103.1	23.5	1.94
Anti Reflectance Fused Silica Flight Sample	30.8	532.2	41.6	102.2	26.1	1.59
Mounting Gasket Silicone Rubber	47.0	532.3	23.4	102.3	27.4	0.85

Silicone 101.5 - 102 eV Binding Energy

SiO2 103 - 103.5 eV Binding Energy

Table 2. XPS analysis data