NASA-CR-205029



**Final Report** 

submitted to

#### NATIONAL AERONAUTICS AND SPACE ADMINISTRATION GEORGE C. MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

March 24, 1997

for Contract NAS8 - 38609

**Delivery Order 136** 

entitled

#### Space Environmental Effects on Thermal Control Coatings

by

Susan K. O'Brien Senior Research Associate and Gary L. Workman Ph.D. Principal Investigator

Materials Processing Laboratory Center for Automation & Robotics University of Alabama in Huntsville Huntsville, Alabama 35899

#### **Table of Contents**

1.0 INTRODUCTION	.1
2.0 NUV IMPACT DAMAGE STUDY	.1
3.0 DISCUSSION OF LABORATORY PORTABLE SPECTROPEEL ECTOMETER SCANS	ICTION
SPECIROREFLECIONETER SCANS	
3.1 FUSED SILICA WINDOW #1	4
3.2 FUSED SILICA WINDOW #2	4
3.3 Pyrex Window #1	5
3.4 Pyrex Window #2	5
3.5 Optical Witness Sample #1	5
3.6 Optical Witness Sample #3	5
3.7 Optical Witness Sample #4	6
3.8 Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-3	6
3.9 Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-7	6
3.10 Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-13	6
3.11 CHROMIC ANODIZED ALUMINUM	7
3.12 2219 Aluminum Sample #3	7
3.13 2219 ALUMINUM SAMPLE #4	7
3.14 CONTROL SAMPLES	7
4.0 TREND DATA	7
5.0 DISCUSSION OF CHANGES IN SAMPLE APPEARANCE	46
6.0 POSSIBLE CAUSES FOR SAMPLE CHANGES	67
7.0 VERIFYING THE INTEGRITY OF THE NUV CHAMBER	68
8.0 CONCLUSIONS	.69

a receiver commence

#### **1.0 Introduction**

The study of long term near ultra-violet (NUV) effects in a vacuum atmosphere, is a crucial element for space applications. NUV radiation causes significant changes in the reflectance of many coatings and types of materials. An ultra high vacuum NUV system was assembled in order to investigate various coatings and materials in this hostile environment. The vacuum is an ion pump that maintains a minimum vacuum in the mid  $10^{-9}$  range. The system has a base pressure of  $10^{-9}$  torr and this base pressure is maintained with the ion pump. The NUV exposure was maintained at 2-3 suns which allows accelerated NUV exposure without overheating the samples. The goal of this test was to maintain an intensity of  $3.4 \times 10^{-2}$  Watts/cm<sup>2</sup> which equals 2.9 NUV suns. An NUV sun is defined as 1.16 Watts/cm<sup>2</sup> integrated over wavelength of 200 - 400 nanometers.

#### 2.0 NUV Impact Damage Study

Monitoring sample reflectance after extended NUV exposure, is very valuable information for new space projects. On January 5, 1995, eight one inch diameter disks and one 2 x 6 plate were placed in the long term NUV vacuum test system, see figure 1. The one inch diameter samples were three Z-93 white diffuse paint discs, three optical witness samples (MgF<sub>2</sub>), and two 2219 aluminum discs. The 2 inch x 6 inch plate was chromic acid anodized aluminum plate. This study had three main purposes, 1) to study the long term NUV effects on materials, 2) determine any contamination effects and 3) determine wavelength dependence of NUV induced damage on materials. This was accomplished by placing Quartz and Pyrex windows over two of the three Z-93 and optical witness samples in order to quantify the NUV effects. The Pyrex window absorbed the NUV photons at wavelengths below 350 nm. Therefore the Pyrex window shielded the underlying sample from NUV photons below 350 nm. Fused silica has eighty percent transmission at 200 nm, therefore the fused silica window allowed the underlying sample to be exposed to the NUV. The Pyrex and the fused silica window also protected the underlying samples from contamination deposition. This enabled a precise method of determining whether the change in solar absorptivity was caused by NUV or contamination. The windows were raised from touching the samples with a 1/16" spacer ring, see figure 2. These specifications are referenced in the final report "Study of the Space Environmental Effects on Spacecraft Engineering Materials" dated May 1, 1995.



Figure 1. Test specimen layout in the sample holder.



Figure 2. Cut away view if the sample holder.

Since the beginning of the study, two different light sources were used and three different bulbs. The first failure of the Hg-Xe bulb occurred in February of 1995. The bulb was replaced and the output adjusted to  $5 \times 10^{-2}$  watts/cm<sup>2</sup> at the sample, which equates to In June of 1995, problems began to arise with the NUV source therefore the system was replaced with a MacPherson lamp. When this lamp was implemented we also added a water filter to the overall system. This water filter aided in the removal of the infrared wavelength from the Hg-Xe output and prevent the samples from experiencing excessive thermal effects. See figure 3 for the new set-up of the system.



Figure 3. System set-up.

It was decided to operate the lamp between two to three NUV suns or  $2.38 \times 10^{-2}$  to  $5.8 \times 10^{-2}$  watts/ cm<sup>2</sup> in order to prevent excessive thermal effects. The lamp system was measured at  $3.358 \times 10^{-2}$  watts\cm<sup>2</sup> using Optronic Laboratories model 752 Spectroradiometer, which equates to approximately 2.9 NUV suns assuming that 1.16 x  $10^{-2}$  equals 1 UV sun. The 752 spectroradiometer scans the wavelengths from 200 to 400 nanometers. The IL1700, scanning from 190 nm to 4.2 microns, measured the output to be  $3.90 \times 10^{-2}$  watts/cm<sup>2</sup>. The In order to periodically check the output of the lamp, a reading was taken on the opposite side of the chamber through the Pyrex viewport. The measurement using the IL1700 was  $1.85 \times 10^{-2}$  watts/cm<sup>2</sup>. Thus knowing this value, the system was checked at various intervals to record and adjust, if necessary, the output intensity of the NUV lamp. Throughout the study numerous power outages occurred, which caused the lamp to go out and at times the ion power supplies to fail.

#### 3.0 Discussion of Laboratory Portable Spectroreflectometer Scans

Periodic reflectance scans were obtained using the Laboratory Portable Spectroreflectometer (LPSR) on the control and exposed samples to measure any changes in the integrity of the samples throughout the study. The samples were exposed for a total time of approximately 28,000 Equivalent Sun Hours (EHS) over a 418 day period. Over that time period, eight reflectance scans were performed. The time schedule of these scans are listed in the table below.

Scan	Date	Days Exposed
Base Line	January 5, 1995	0 Days
1 <sup>st</sup> Scan	February 16, 1995	42 Days of Exposure - 6 Weeks
2 <sup>nd</sup> Scan	April 3, 1995	88 Days of Exposure ~ 12.5 Weeks
3 <sup>rd</sup> Scan	August 3, 1995	123 Days of Exposure ~ 17.5 Weeks
4 <sup>th</sup> Scan	September 6, 1995	155 Days of Exposure ~ 22 Weeks

5 <sup>th</sup> Scan	January 19, 1996	290 Days of Exposure ~ 41 Weeks
6 <sup>th</sup> Scan	May 8, 1996	398 Days of Exposure ~ 57 Weeks
7 <sup>th</sup> Scan	June 17, 1996	418 Days of Exposure ~ 60 Weeks

When the samples were removed from the system, the gate valve was closed which isolated the test chamber from the ion pump and the test chamber was returned to atmosphere using dry nitrogen as a repressurization gas. Once at atmosphere, the Pyrex viewport was removed and the sample holder was taken out of the system. A complete step-by- step description of the procedures for the sample measurements can be found in the section Probable Causes for Sample Changes. A total of 15 sample scans were obtained per scan date. The windows that cover the Z-93 paint, the optical witness samples and the control windows were scanned using the control optical witness sample #2 behind the window. See figure 4. A black tube was placed around the sample set-up during scanning to insure that no stray light entered the aperture of the LPSR.



Figure 4. LPSR measurement set-up for the windows.

Five control samples were scanned prior to the exposed samples to verify continuity. Below is a brief description of the history of each sample that was exposed to NUV radiation.

#### 3.1 Fused Silica Window #1

The fused silica window #1 protected optical witness sample #4 during NUV exposure. No changes were found in the LPSR scans until January 19,1996, after 290 days of exposure. On this scan we can see a drastic decrease in the spectral reflectance beginning at 550 nanometers. At 250 nanometers the spectral reflectance dropped from 0.85 to 0.73, a 14% decrease. Then the spectral reflectance dropped again on June 16 from 0.73 to 0.67, another 8% drop. Slight increases, around 1% between each scan, in the spectral reflectance occurred in the 1250 to 2500 nanometer range until the final scan on June 17, 1996 when the spectral reflectance dropped across the whole spectrum. But these slight variations in the spectral reflectance can be accounted for in the accuracy on the LPSR, being only  $\pm 1$ %. Solar alpha jumped from 0.11 on May 8<sup>th</sup> to 0.13 on June 17<sup>th</sup>. And an overall increase, when comparing the baseline to the final scan, from 0.10 to 0.13.

#### 3.2 Fused Silica Window #2

Fused silica window #2 behaved in a similar manner to Fused silica window #1. This window protected Z-93 #B169-13 white diffuse paint sample during the NUV exposure. There was a significant decrease in the reflectance in the region from 250 to 600 nanometers. And once again there was a decrease in the reflectance across the entire range on the June 17, 1996 scan. Solar alpha jumped from 0.11 to 0.13. And an overall increase, when comparing the baseline to the final scan, from 0.10 to 0.13.

#### 3.3 Pyrex Window #1

Pyrex window #1 covered optical witness sample #3 during NUV exposure. The Pyrex window #1 showed a drop between the baseline scan and all following runs from 275 to 450 nanometers. Then a drop in reflectance from 475 to 750 nanometers when comparing the final run on June 17, 1996 and the previous runs. One interesting thing to note is that at 275 nanometers the reflectance increased on the last three runs performed January 19<sup>th</sup>, May 8<sup>th</sup>, and June 17<sup>th</sup>. Solar alpha jumped from 0.15 on scan performed May 8 to 0.16 when taken June 17<sup>th</sup>. And an overall increase, when comparing the baseline to the final scan, from 0.14 to 0.16.

#### 3.4 Pyrex Window #2

Pyrex window #2 protected the Z-93 #B169-7 white diffuse paint sample during the NUV exposure. This window #2 behaved in the same manner as Pyrex window #1 with the baseline having the highest spectral reflectance in the region from 275 to 450 nanometers then all following runs drop in this range. It is also seen that the spectral reflectance rises on the last three runs from 250 to 275 nanometers. The solar alpha jumped from 0.15 to 0.16 when comparing runs on May 8<sup>th</sup> and June 17<sup>th</sup>. There was an overall increase with the baseline solar alpha of 0.13 and final alpha of 0.16.

#### 3.5 Optical Witness Sample #1

Optical witness sample #1 was unprotected during the entire NUV exposure study meaning it had full exposure to NUV and any possible contamination. In evaluating the LPSR scans, the spectral reflectance remained constant for all scans until the January 19<sup>th</sup> scan. On this scan a drastic decrease occurred at approximately 450 nanometers. The reflectance dropped from an average of 0.86 to 0.64 on the January 19<sup>th</sup> scan and continued to drop to a final reflectance of 0.61 on June 17<sup>th</sup>. Only minimal changes were seen in the solar alpha which started at 0.093 at the baseline and ended at 0.098 on June 17<sup>th</sup>.

#### 3.6 Optical Witness Sample #3

Optical witness sample #3 was protected with a Pyrex window during the entire NUV exposure study which blocked the NUV rays. No changes were seen in the spectral reflectance for the optical witness sample until the last run on June 17<sup>th</sup>. On the June 17<sup>th</sup> run, the reflectance dropped starting at 375 nanometers and continued to drop until 250

nanometers. At 250 nanometers the reflectance dropped from an average of 0.877 to 0.836. A slight decrease of the solar alpha occurred over the entire study duration. Solar alpha went from a baseline reading of 0.094 to 0.089 on June  $17^{\text{th}}$ .

#### 3.7 Optical Witness Sample #4

Optical witness sample #4 was protected with fused silica window #1 during exposure. This sample behaved in the same manner as optical witness sample #3. There was a decreasing reflectance starting at 375 nanometers and continued to drop until 250 nanometers. Solar alpha went from a baseline of 0.093 to 0.091 on the June 17<sup>th</sup> scan.

#### 3.8 Z-93 White Diffuse Paint Sample #B169-3

Z-93 sample #B169-3 was not protected during the NUV exposure study. The spectral reflectance decreased with each run in the 425 to 750 nanometer range. The most drastic decrease occurred between the baseline and the first scan on February 16<sup>th</sup>. An interesting thing to note is that the reflectance went back up slightly on the last run taken on June 17<sup>th</sup>. There were slight decreases in the spectral reflectance between each scan in the 1750 to 2500 nanometer range. When looking at the solar alpha, alpha continued to rise until the last scan on June 17<sup>th</sup> where it dropped from 0.182 to 0.175. The overall alpha changed from 0.157 on the baseline scan to 0.175 on the June 17<sup>th</sup> scan, the last scan performed.

#### 3.9 Z-93 White Diffuse Paint Sample #B169-7

This sample was protected with Pyrex window #2 during the entire NUV exposure. This sample behaved in a different manner in the 425 to 750 nanometer region. There was no decrease in reflectance in this range. There were slight decreases in the spectral reflectance between each scan in the 1750 to 2500 nanometer range, which was similar to sample #B169-3. The solar alpha remained stable until the last two scans, scans May 8<sup>th</sup> and June 17<sup>th</sup>, where alpha increased slightly. The was not a decrease in alpha on the last scan as seen on sample #B169-3, the unprotected sample. Solar alpha went from a baseline of 0.155 to a final value of 0.160, which was significantly less of a change.

#### 3.10 Z-93 White Diffuse Paint Sample #B169-13

Z-93 sample #B169-13, which was protected with fused silica window #2, scans were similar to sample #B169-3. There was a decrease between each scan in both the 425 to 750 nanometer range and the 1750 to 2500 nanometer range. The solar alpha behaved slightly different in that there was not a rise in alpha on the final run on June  $17^{\text{th}}$ . Alpha increased throughout the entire run, beginning at 0.157 for the baseline and ending at 0.183 for the final scan.

#### 3.11 Chromic Anodized Aluminum

Three scans were taken on the chromic anodized aluminum plate, a top, center and bottom scan of the plate, to ensure an overall reading. The three scans behaved in a similar manner, therefore we will only talk about the center scan. As the amount of NUV exposure increased, the scans progressed to flatten in the region below 700 nanometers. Solar alpha increased slightly from the baseline scan until 88 days of NUV exposure. After 88 days of exposure alpha began to fall until 290 days of exposure, then rose sharply at 418 days. The overall solar alpha decreased from the beginning baseline reading of 0.364 to 0.354 for the final scan.

#### 3.12 2219 Aluminum Sample #3

The 2219 aluminum sample #3 was unprotected throughout the NUV exposure. The spectral reflectance remained stable until January 19, 1996, when the reflectance dropped beginning at 425 nanometers and continuing to fall to 250 nanometers. The solar alpha remained steady. Solar alpha went from a baseline of 0.375 to a final value of 0.379, no change .

#### 3.13 2219 Aluminum Sample #4

Sample #4 was also unprotected during the study and behaved in the same manner as sample #3. The was a decrease in reflectance after 425 nanometers. There was a slight increase in solar alpha. A baseline alpha of 0.322 and a n ending alpha of 0.331, overall the line in fairly flat with minor changes.

#### **3.14 Control Samples**

No changes were found in the control sample scans.

#### 4.0 Trend Data

Trends in Solar Alpha

Sample	Baseline Alpha	Final Alpha	Delta
Fused Silica Window #1	0.104	0.127	0.023
Fused Silica Window #2	0.103	0.131	0.028
Pyrex Window #1	0.137	0.161	0.024
Pyrex Window #2	0.133	0.163	0.030
Optical Witness Sample #1	0.093	0.098	0.005
Optical Witness Sample #3	0.094	0.089	-0.005
Optical Witness Sample #4	0.093	0.091	-0.002
Z-93 #B169-3	0.157	0.175	0.018
Z-93 #B169-7	0.155	0.160	0.005
Z-93 #B169-13	0.157	0.183	0.026

CAA Top Scan	0.362	0.357	-0.005
CAA Center Scan	0.364	0.354	-0.010
2219 Aluminum #3	0.375	0.379	0.004
2219 Aluminum #4	0.322	0.331	0.009

The spectral reflectance and solar alpha graphs for the control and experimental samples are on the following pages, figures 5-41.





1. S. S. S. S.

영국 문화 관계 전문 문제

File: WINDOWS.XLS Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



Figure 6. Fused silica window #1 solar alpha profile.





11

File: WINDOWS.XLS

Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

CHANGE IN SOLAR ALPHA FOR FUSED SILICA WINDOW #2 FUSED SILICA WINDOW #2 PROTECTED Z-93 B-169-13 SAMPLE DURING UV EXPOSURE SAMPLE WAS EXPOSED FOR 418 DAYS TO UV RADIATION WHILE UNDER VACUUM 0.25 0.20 SOLAR ALPHA 0.15 0.10 0.05 0.00 10,000 12,500 15,000 17,500 20,000 22,500 25,000 27,500 30,000 5,000 7,500 2,500 0 **EQUIVALENT SUN HOURS** 

Figure 8. Fused silica window #2 solar alpha profile.

17 6 You 19 19 19 19 19 19 19 20 19 20 20 19 19

File: WINDOWS.XLS Data Taken:1/5/95,2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



# PYREX WINDOW #1 - LPSR DATA

Figure 9. Pyrex window #1 spectral reflectance scans.

13

> File: WINDOWS.XLS Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96





File: WINDOWS.XLS Data Taken:1/5/95,2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



Figure 11. Pyrex window #2 spectral reflectance scans.

File: WINDOWS.XLS

Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



CHANGE IN SOLAR ALPHA FOR PYREX WINDOW #2

Figure 12. Pyrex window #2 solar alpha profile.

となった ほうしき ほう おものもの



17

Data Taken: 1/5/95, 2/16/95, 4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



CHANGE IN SOLAR ALPHA FOR OPTICAL WITNESS SAMPLE #1

والإركاري المتعارف ومتحج المعتور فالها المتعارين والروا





Data Taken: 1/5/95, 2/16/95, 4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96



# CHANGE IN SOLAR ALPHA FOR OPTICAL WITNESS SAMPLE #3

Figure 16. Optical witness sample #3 solar alpha profile.



Figure 17. Optical witness sample #4 spectral reflectance scans.

21

File: OWS.XLS Data Taken: 1/5/95, 2/16/95, 4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_23_Figure_0.jpeg)

Figure 18. Optical witness sample #4 solar alpha profile.

22

File: OWS.XLS

![](_page_24_Figure_0.jpeg)

![](_page_24_Figure_1.jpeg)

Data Taken: 1/05/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_25_Figure_0.jpeg)

CHVNCE IN SOLAR ALPHA FOR Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-3

Figure 20. Z-93 sample #B169-3 solar alpha profile. 24

· 동네 그는 것 같이 가지 않는 것은 것 같은 것 같이 있다.

![](_page_26_Figure_0.jpeg)

# Figure 21. Z-93 sample #B169-7 spectral reflectance scans.

영화 동물 문화 이 나는

. . . . . .

Data Taken: 1/05/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

#### **EXAMPLE EXPOSED FOR 418 DAYS TO UV #B169-7 WAS PROTECTED WITH PYREX WINDOW #2 DURING UV EXPOSURE CHANGE IN SOLAR ALPHA FOR Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-7**

المراجع المراجع ومحمدتها ومنابع المراجع المراجع المراجع والمراجع والمراجع والمراجع

![](_page_27_Figure_1.jpeg)

Figure 22. Z-93 sample #B169-7 solar alpha profile.

![](_page_28_Figure_0.jpeg)

![](_page_28_Figure_1.jpeg)

말했는 것 않았는 것 같아? 그는 것 같아요.

File: Z93.XLS Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_29_Figure_0.jpeg)

# CHANGE IN SOLAR ALPHA FOR Z-93 WHITE DIFFUSE PAINT SAMPLE #B169-13

**EQUIVALENT SUN HOURS** 

Figure 24. Z-93 sample #B169-13 solar alpha profile.

1. **1**. (....)

![](_page_30_Figure_0.jpeg)

#### **CHROMIC ANODIZED ALUMINUM SAMPLE II-6**

29

Data Taken: 1/5/95, 2/16/95,4/3/95, 8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_31_Figure_0.jpeg)

ΕΦΠΙΛΥΓΕΛΙ 2ΠΛ ΗΟΠΒ2

CHANGE IN SOLAR ALPHA FOR CHROMIC ANODIZED ALUMINUM II-6

30

Figure 26. Chromic anodized aluminum sample II-6 solar alpha profile.

法公司 化乙基苯基乙基苯基乙基苯乙基

![](_page_32_Figure_0.jpeg)

![](_page_32_Figure_1.jpeg)

31

File: 2219ALU.XLS

------

![](_page_33_Figure_1.jpeg)

![](_page_33_Figure_2.jpeg)

32

SOLAR ALPHA

0.50

0.45

0.40

#### CHANGE IN SOLAR ALPHA FOR 2219 ALUMINUM SAMPLE #3 2219 AL WAS NOT PROTECTED WITH A WINDOW DURING EXPOSURE SAMPLE WAS EXPOSED FOR 418 DAYS TO UV RADIATION WHILE UNDER VACUUM

2500 42 Days Exp. @ 3 UV Suns
42 Days Exp. @ 3 UV Suns
90 Days @ 3 UV Suns & 33 Days @ 2.89 UV Suns
90 Days @ 3 UV Suns & 65 Days @ 2.89 UV Suns
90 Days @ 3 UV Suns & 65 Days @ 2.75 UV Suns
90 Days @ 3 UV Suns & 308 Days @ 2.75 UV Suns
90 Days @ 3 UV Suns & 328 Days @ 2.75 UV Suns BASELINE VS 418 DAYS OF UV EXPOSURE, ALPHA=0.322 BEFORE VS 0.331 AFTER EXPOSURE 2250 2000 2219 AL #4 WAS NOT PROTECTED WITH A WINDOW DURING UV EXPOSURE WAVELENGTH IN NANOMETERS (nm) 1750 Baseline **2219 ALUMINUM SAMPLE #4** 1500 1250 1000 750 500 250 0.0 0.5 0.3 0.2 1.0 0.6 0.4 0.1 0.9 0.8 0.7 **SPECTRAL REFLECTANCE** 

File: 2219ALU.XLS ביוביי אותבותב אומותב אומות אומות אומות אומות אומות אומות א

Figure 29. 2219 aluminum sample #4 spectral reflectance scans.

![](_page_35_Figure_0.jpeg)

Figure 30. 2219 aluminum sample #4 solar alpha profile.

File: 2219ALU.XLS Data Taken:1/5/95,2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_36_Figure_0.jpeg)

#### FUSED SILICA CONTROL WINDOW #3 UV EXPOSURE STUDY - LPSR DATA 418 DAYS SINCE INITIAL SCAN

35

1.1

Figure 31. Fused silica control window #3 spectral reflectance scans.

Data Taken: 1/5/95, 2/16/95,4/3/95i,8/3/95,9/6/95,1/19/96i,5/8/96,6/17/96

#### 418 DVAS SINCE INITIAL SCAN CHAINGE IN SOLAR ALPHA FOR CONTROL FUSED SILICA WINDOW #3

![](_page_37_Figure_1.jpeg)

36

Figure 32. Fused silica control window #3 solar alpha profile.

#### **\*18 DVAS SINCE INILIAL SCAN OPTICAL WITNESS CONTROL SAMPLE #2 UV EXPOSURE STUDY - LPSR DATA**

![](_page_38_Figure_1.jpeg)

Data Taken: 1/5/95, 2/16/95, 4/3/95, 8/3/95, 9/6/95, 1/19/96, 5/8/96, 6/17/96

37

Figure 33. Optical witness control sample #2 spectral reflectance scans.

# 418 DV ASS SINCE INITIAL SCAN CHANGE IN SOLAR ALPHA FOR CONTROL OPTICAL WITNESS SAMPLE #2

![](_page_39_Figure_1.jpeg)

![](_page_40_Figure_0.jpeg)

![](_page_40_Figure_1.jpeg)

4. 4. 1 (1) (1) (1) (2) (4)

Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

방법 수는 그는 그 그는 것을 가지 않는 것을 했다.

#### 418 DVAS EBOW INILIVT SCVN CHVNCE IN SOLAR ALPHA FOR Z-93 #B169-1 CONTROL SAMPLE

![](_page_41_Figure_1.jpeg)

Data Taken:1/5/95,2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

![](_page_42_Figure_0.jpeg)

![](_page_42_Figure_1.jpeg)

41

Data Taken: 1/5/95. 2/16/95.4/3/95.8/3/95.9/6/95.1/19/96.5/8/96.6/17/96

에는 이번 가슴에 있는 것 같은 것은 것이 같은 것이 있다. 가슴이 있는 것이 가지 않는 것이 있는 것이 있는 것이 있다. 같은 것이 같은 것이 같은 것이 같은 것이 같은 것이 같은 것이 있는 것이 있다. 것이 같은 것이 없다. 것이 있 같은 것이 없다. 것이 없이

![](_page_43_Figure_0.jpeg)

![](_page_43_Figure_1.jpeg)

![](_page_44_Figure_0.jpeg)

#### 2219 ALUMINUM CONTROL SAMPLE #5 UV EXPOSURE STUDY - LPSR DATA 418 DAYS SINCE INITIAL SCAN

43

<u>د</u> .

Data Taken: 1/5/95, 2/16/95,4/3/95,8/3/95,9/6/95,1/19/96,5/8/96,6/17/96

#### 418 DVAS SINCE INILIVT SCVN CHVNCE IN SOFVE VFLHV EOE 5516 VFNMINNM CONLEGT 89WLFE #2

![](_page_45_Figure_1.jpeg)

Figure 40. 2219 aluminum control sample #5 solar alpha profile.

44

File: CONTROLS AND THE ALANCE ALANCE FROM FILE .

![](_page_46_Figure_0.jpeg)

![](_page_46_Figure_1.jpeg)

45

Figure 41. Trends in solar alpha for the thirteen test samples.

File: Solar Alpha.XLS

- - - - AIFINE NIADINE AININE NININE NIDINE AIANINE EININE EININE EININE

#### 5.0 Discussion of Changes in Sample Appearance

On May 8<sup>th</sup>, 1996, a change was noticed in the appearance of some of the samples. There appeared to be a darkening effect on the windows that protected the samples. Changes were also seen on the chromic anodized aluminum (CAA). The CAA was darker on the exposed area and a speckle pattern was concentrated in the center of the sample. The speckles appeared to be a type of pitting effect. Due to these changes, transmission spectra were performed on the windows to measure any possible changes. Five spectra were taken, one on each exposed window and a quartz window that was atomic oxygen cleaned and one that was only cleaned with ethyl alcohol. There was an obvious decrease in transmission first noticeable around 325 nanometers and ending at 1600 nanometers for both Pyrex windows. The quartz windows saw a large decrease in transmission around 200 nanometers continuing until 825 nanometers. The scans were taken from 200 to 1000 nanometers. The transmission scans can be found on pages 47-51, figures 42-46 The samples were returned to the NUV chamber after the LPSR scans were performed.

On May 31<sup>st</sup>, the samples were removed to take another LPSR reading on the samples to see if any further degradation had occurred to the samples. The LPSR was not behaving properly and was taken in for servicing. Irregularities were found on the sphere surface of the LPSR and the sphere was replaced with a new integrating sphere. LPSR was operational again by June 17<sup>th</sup>, and the samples were removed for scanning. A decreased spectral reflectance was found in the fused silica windows, optical witness samples and 2219 aluminum samples when comparing to the May 8th scan. The solar alpha increased for the fused silica windows, Pyrex windows, and CAA. It decreased for the Z-93 #B169-3 sample and remained constant on the 2219 aluminum, Z-93 #B169-7 and #B169-13 samples when compared to the May 8th scan. Upon visually inspecting the Z-93 #B169-3 unprotected sample, a light brown discoloration was noticed over the entire surface with a higher concentration on 1/4 of the sample. A brownish discoloration was also seen on the entire Z-93 #B169-13 fused silica protected sample. No other visual changes were found since the May 8th scan in the other samples. Standard photographs were taken of all the samples to documented the visual changes found in some of the samples. The samples were scanned to attain a digital format of the pictures for reporting purposes. The scanned samples are on pages 52-58, figures 47-59. There are some discrepancies in the true colors of the samples. The main thing we want to illustrate is the darkening effect seen on some of the Z-93 samples. In figure 54, the browning effect of the Z-93 #B169-3 sample is found in the picture. This effect is apparent in the bottom portion of the sample where there is a interface line approximately a 1/16" from the edge of the sample. This interface is where the sample was partially covered by the lip of the sample holder, therefore the outer edge of the sample did not see the NUV effects. The optical witness samples appear white in the photograph because it is a total reflective surface. On the chromic anodized aluminum sample the pitting of the surface is not noticeable in the scanned image but the darkening effect is visible. .It should also be noted that the browning effect is degrading over time. The Z-93 paint samples were examined under a microscope to see if there were any abnormalities in the surface. Upon examination, small fibers, apparent metal fragments and possible pitting in the paint

were found embedded in the paint. Therefore this needs to be considered when we talk about causes of the darkening effect found on the Z-93 samples. Two scanning electron micrographs were taken at 500X of two of the sample anomalies on the Z-93 samples, including one of the fibers and one of the metal flakes. Four micrographs were taken at 100X magnification of the metal fragments, fiber and fuzz found imbedded in the Z-93 B169-3 and B169-1 samples. These anomalies were found in all Z-93 B169 samples including the control sample. The SEM images, photoelectron spectra and micrographs of the flaws in the Z-93 paint samples are on pages 58-65, figures 60-69.

# I# wobniw x9rgan Transmission Scan

![](_page_49_Figure_1.jpeg)

Figure 42. Transmission scan for Pyrex window #1.

48

File: PYREX.XLS Data Taken: 5/8/96

![](_page_50_Figure_0.jpeg)

**Transmission Scan** 

![](_page_50_Figure_2.jpeg)

49

Figure 43. Transmission scan for Pyrex window #2.

File: PYREX.XLS Data Taken: 5/8/96

# I# wobniW soilis bosuA

## neol noissiment

![](_page_51_Figure_2.jpeg)

Figure 44. Transmission scan for fused silica window #1.

# **Fused Silica Window #2**

**Transmission Scan** 

![](_page_52_Figure_2.jpeg)

Figure 45. Fused silica window #2 transmissions scan.

![](_page_53_Figure_0.jpeg)

![](_page_53_Figure_1.jpeg)

## A.O. and Alcohol Cleaned

![](_page_53_Figure_3.jpeg)

![](_page_54_Figure_0.jpeg)

Figure 47. Fused silica window #1 protected optical witness sample #4.

![](_page_54_Picture_2.jpeg)

Figure 48. Fused silica window #2 protected Z93 #B169-13 sample.

![](_page_55_Figure_0.jpeg)

Figure 50. Pyrex window #2 protected Z93 #B169-7 sample.

![](_page_56_Picture_0.jpeg)

Figure 51. Unprotected optical witness sample #1.

![](_page_56_Picture_2.jpeg)

Figure 52. Optical witness sample #3protercted by Pyrex window #1.

![](_page_57_Picture_0.jpeg)

Figure 53. Optical witness sample #4 protected by fused silica window #1.

![](_page_57_Picture_2.jpeg)

Figure 54. Unprotected Z93 #B169-3 sample.

Ł

![](_page_58_Picture_0.jpeg)

Figure 55. Z93 #B169-7 sample protected by Pyrex window #2.

![](_page_58_Picture_2.jpeg)

Figure 56. Z93 #B169-13 protected by fused silica window #2.

![](_page_59_Picture_0.jpeg)

Figure 57. Unprotected 2219 aluminum sample #3

![](_page_59_Picture_2.jpeg)

Figure 58. Unprotected 2219 aluminum sample #4.

![](_page_60_Figure_0.jpeg)

Figure 59. Unprotected chromic anodized aluminum sample II-6C.

![](_page_60_Picture_2.jpeg)

Figure 60. SEM photo of one of the Z-93 B169-3 imbedded fibers.

![](_page_61_Picture_0.jpeg)

Figure 61. SEM photo of one of the Z-93 B169-3 metal flakes.

![](_page_62_Figure_0.jpeg)

![](_page_63_Figure_0.jpeg)

Detector Resolution: 148.50 eV Take-off angle= 40.00

Figure 63. Photoelectron spectra of Z-93 #B169-3 imbedded particle.

![](_page_64_Figure_0.jpeg)

![](_page_65_Figure_0.jpeg)

Figure 65. Photoelectron spectra of Z-93 #B169-3 background scan.

![](_page_66_Picture_0.jpeg)

Figure 66. Z-93 #B169-1 100X micrograph of imbedded metal flakes.

![](_page_66_Picture_2.jpeg)

Figure 67. Z-93 #B169-1 100X micrograph of imbedded fuzz.

![](_page_67_Picture_0.jpeg)

Figure 68. Z-93 #B169-1 100X micrograph of imbedded fiber.

![](_page_67_Picture_2.jpeg)

Figure 69. Z-93 #B169-3 100X micrograph of imbedded metal flake.

#### 6.0 Possible Causes for Sample Changes

When we look at all of the data we have obtained on the samples, the spectral reflectance scans, transmission scans and visual inspections, the question arises, what caused the sudden change in spectral reflectance after approximately 19,500 Equivalent Sun Hours? In order to answer this question we must look at everything that happened to the system on and prior to the January 19<sup>th</sup> scan. It appears from the behavior of the spectral reflectance scans that the samples have been contaminated. We can not exactly determine the true source of the contamination without extensive testing of the samples. Some of the possible contamination sources are 1) the material that was imbedded in the Z-93 white diffuse paint, see figures 68 and 69, 2) the method by which the samples were removed, 3) numerous power outages throughout the study including while NASA was on furlough, and 4) any other possible abnormalities that occurred during the test.

The first item we will discuss is the fact that the Z-93 series #B169 samples were prepared in a non-clean room facility. On inspection of the samples under a microscope small pieces of fiber and flakes of metal fragments appeared on the surface of the paint and imbedded in the paint itself. These items were most likely adhered to the surface when the white diffuse paint was sprayed onto the samples. The question is if this caused the discoloration of the sample and the changes in the reflectance, why did it take so long to appear. This is still not known at this time and needs further investigation until we can exactly pin point if this truly was the cause of the changes in the samples. The second issue to address is the method used for taking the LPSR measurements. During this procedure there are a number of unknowns that could be introduced into the system. Although we are extremely cautious, there are factors to consider. When the samples are removed from the chamber, the following procedure is used.

1) The gate valve is closed to isolate the test chamber from the ion pumps.

2) The test chamber is repressurized with nitrogen obtained from a liquid nitrogen dewier and bought up to atmospheric pressure. This is repressurized by using a Teflon tube that is attached to the test chamber via a needle valve and the other end is typically inserted into the gas outlet of the dewier. But the hose has been taped to the outlet port of the dewier using electrical tape and duct tape at one point in time. It is not clearly known at which point in time this tape was applied. It is doubtful that this could have been the cause of the contamination because of the large change in the spectral reflectance of the optical witness samples after the chamber was glow discharged. But it needs to be noted.

3) Once the test chamber is at atmosphere, the Pyrex viewport is removed. The sample holder is removed using latex powder free Class 100 clean room gloves. The sample holder is then wrapped in aluminum foil. The side where the Pyrex viewport was removed is then covered with aluminum foil to prevent air born particulates from getting into the system.

4) The sample holder is then taken to the LPSR for scanning. Before the scan is performed the measurement aperture is wiped using an alpha wipe dampened with ethyl alcohol contained in a Teflon bottle to remove any possible contamination.

5) First the control samples are scanned to insure repeatability of the LPSR. Once this is complete, the sample holder is unwrapped from the aluminum foil and the samples are removed one at a time and scanned. The samples are removed using Teflon or stainless tweezers cleaned in ethyl alcohol using an alpha wipe. Once all of the samples are scanned the sample holder is taken back to the chamber.

6) The sample holder is placed back into the chamber, the copper gasket is replaced and the flange is bolted back into place. The chamber is then purged several times with nitrogen and the contamination free roughing pump is allowed to pump on the system.

7) Once the vacuum in the test chamber is below 10 millitorr, the ion pumps are switched over to the start mode and the gate valve is opened.

8) The ion pumps are allowed to pump on the system overnight, so if there is any contaminants they would be pulled down into the ion pumps. The NUV light source is turned on the following day.

The third item that needs to be discussed is the numerous power outages throughout the study. Most of the outages that occurred did not seem to have any effect on the system. But these outages occurred while MSFC employees were still at work and the NUV chamber was turned back on in a timely matter. There was one power outage that occurred in December during the furlough and the system was down for an undetermined amount of time. The scan taken in January was the first time the large decrease in the spectral reflectance occurred. It is not known if something might have happened while the system was down in December.

Some other miscellaneous items that need to be mentioned is the gate valve was not always functioning properly. Sometimes the valve got stuck in the open position and was cycled numerous times until it closed. The gate valve also leaked oil through one of the seals outside of the unit. Another item to mention is servicing of the ion pump power supply.

#### 7.0 Verifying the Integrity of the NUV Chamber

Due to leaks in the vacuum chamber and possible contamination the study was terminated. After removal of the samples glow discharge was done on the entire chamber. The glow discharge was run for approximately 40 hours. On completion of the glow discharge a new optical witness sample was loaded into the chamber. The sample was left in the dark chamber, no NUV source was turned on, for one week. Upon removal of the sample a scan was performed with the LSPR. The sample showed extreme signs of contamination. After completing this test, it appears that there is a large amount of contamination, possibly a insect or something of this magnitude. Efforts will continue to clean the system and if there is no success, the system will be completely disassembled, thoroughly cleaned and re-assembled.

#### 8.0 Conclusions

- Although contamination caused an early conclusion to this study, we still attained very valuable information concerning NUV radiation effects on various surfaces. We attained not only information for a duration of 10,770 equivalent sun hours of contamination free NUV radiation but also the effects an unknown contaminant can have on these particular surfaces.
- Continued efforts on determining the source of the contamination or possibly the type of contamination would be valuable information.
- Optimization of the procedure for taking the LPSR measurements to insure no contamination or modifying the existing UV system .
- We now know that after 10,770 equivalent sun hours there was little to no change in the optical witness samples and 2219 aluminum samples. There were changes in the Z-93 white diffuse paint in the 425 to 825 nanometer range as well as the 1250 to 2500 nanometer range. And the chromic anodized aluminum saw a flattening effect in the spectral reflectance in the 250 to 500 nanometer range.
- Further studies need to continue on the effects of NUV damage, so that we might better understand the effects NUV radiation has on optical surfaces when exposed beyond 10,770 equivalent sum hours.
- Future tests should also include the implementation of a sample cooling system to insure no thermal effects on the samples during NUV exposure.