FINAL REPORT

SURFACE EVALUATION OF UV-DEGRADED CONTAMINATION

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Introduction

Three different areas of work have been accomplished under this contract: 1) contamination testing and evaluation, 2) UV irradiation testing, and 3) surface evaluation testing. Contamination testing was generally performed in the *In-Situ* Contamination Effects Facility at Marshall Space Flight Center (MSFC). UV irradiation testing was also performed primarily at MSFC, utilizing facilities there. Finally, the surface evaluation was done at facilities at UAH Center for Applied Optics.

Contamination Testing

At this time the only contamination testing performed has been on Solithane 113. This is a result of the effects of the outgassing of Solithane in a vacuum at the test temperatures upon the test chamber. After the first test of Solithane, it took a period of three months to be able to achieve the same level of vacuum as had been achieved before the test. After the second test, it took four months to come near the same level of vacuum.

Two tests were performed on Solithane 113. Although the first test was performed during the previous contract, the analysis of the results had not been completed at the writing of the final report, so the results are included herein. Under the general label of Rob Series 5, the two tests have been labeled Run 1 and run 2.

The tests utilized two mirror stands. The primary mirror stand is in the beam path of the VUV beam, and reflectance data is taken during the test. The secondary mirror stand holds four mirrors, and they are only exposed to the contamination. Both stands are cooled, though the secondary stand is indirectly cooled and it does not reach the same temperature of the primary mirror stand. The objective temperature for the primary mirror and the contaminant stand was -5° C and 60° C, respectively.

Run	Mirror	Duration	Total TQCM Change	% Specular Change
1	R4	6.3 hrs	na	-22.4%
2	B-7	24.5 hrs	92 Hz	5.0%

There were some puzzling results from the second test. There was little change in the TQCM frequency, and a positive change in percent specular reflectance, but there was a visible contaminant upon the mirror's surface. This visible contamination was a blue haze, and showed up, to a lesser degree, on the four secondary mirrors.

Some FTIR analysis was done on the contamination found on the mirror R4 from the first test. The possible chemicals found within the contamination are some phenols, large indications of carbon-hydrogen pairs, and some unsaturated ester bonds.

Continuing Efforts

In recent months, the condition of the *In-Situ* Facility has not been up to the standards needed for good contamination testing, so a system refurbishment was started. The purpose was to determine what could be done to increase the usefulness of the system, and to decrease the down time between tests.

This was started with the realization that the chamber's ability to achieve the proper pressure levels needed to be determined. To do this, all the electrical and optical components of the facility were removed from the chamber to a clean room. The chamber was then cleaned properly and it began a series of vacuum bakeouts. At this time, the current pressure level is at approximately 7×10^{-7} torr which is within the target range.

The next tasks will be to put the stage with the optical and electrical components back into the chamber. Before this is done, various new coolant lines need to be constructed and the alignment of the optical system needs to be checked for the problems in the scattered light detectors.

To increase the utility of the facility, the Automatic Contamination Evaluation (ACE) needs to be installed in the chamber. This will allow for two simultaneous tests of a contaminant in the same environment. To achieve this, there will need to be several modifications made to the chamber.

Contamination Testing Conclusion

When these items have been completed, the *In-Situ* Facility will be ready for more contamination testing. The scattered light detectors should once again provide information about the contaminant, and the ACE is expected to give a good comparison to the other data.

UV Irradiation Testing

UV irradiation testing has been done in two chambers at MSFC, viz., (1) the *In-Situ* Facility and (2) a small photodeposition chamber that was adapted for use as an irradiation test site. Most of the effort was in the tests done in the photodeposition chamber.

In-Situ Facility

The *In-Situ* Facility was used in one UV irradiation test. This test was designed simply to determine if prolonged exposure to VUV radiation would induce any chemical change on a contaminated mirror.

Mirror 16-91 has been contaminated as a secondary mirror in Run 1 of the Solithane 113 Tests. For the test, the filter normally used in the Facility was replaced with a MgF_2 window to protect the lamp from any contamination and to ensure that the proper wavelength VUV would be transmitted.

The test ran for 116 hours. When the mirror and window were removed from the chamber, the mirror looked to be less contaminated, and there was a noticeable contamination on the window. There did not appear to be any chemical change in the mirror's contamination. Analysis under a microscope showed that there was less contamination than had been there previously.

Photodeposition Chamber

Two types of tests were done in the photodeposition chamber. The first test was similar to that done in the *In-Situ* Facility. the other tests were set up differently, but were geared towards the same end.

First Test

This test used a Xenon lamp with a filter as the radiation source. The filter characteristics were as follows: peak wavelength 2600 Å, peak transmission 45%, and bandwidth 400 Å.

Mirror R4, the primary mirror from the first Solithane test, was exposed. The test lasted for 214 hours. As in the other test, the amount of contamination on the test mirror decreased during the test.

Second Test

The UV irradiation test site has been designed for the testing of materials to ultraviolet radiation exposure in a vacuum environment. The types of materials that will be tested include seal materials, paint samples, and contaminated mirrors. The system is capable of reaching the 10^{7} torr range and of maintaining a constant sample temperature through coolant lines. Currently, it is configured to utilize high pressure arc lamps as a UV source and a microwave cavity lamp as a VUV source. A water filter is in place to filter out any heat and infrared radiation. Also, a grating has been used to spread out the spectrum of the arc lamp's beam.

Test Report

To date, three different materials have been tested in the chamber. A silicone rubber, S383 type, anodized aluminum, and some doped polystyrene. We have tried several different configurations in the chamber to irradiate our samples, but had little success. The following paragraphs will detail the different test configurations and results.

Anodized Aluminum

The first test done was with anodized aluminum. The anodized aluminum should fade with an exposure to certain levels of UV radiation. The arc lamp was the only source of UV, and was directed through the water filter towards the grating. In this original configuration, the grating was placed on the opposite wall of the chamber, so as to get the largest possible path length. The grating is designed to provide a focussed spectrum along an arc at a radius

of 0.5 m, but the chamber does not have that amount of space, so we came as close as possible.

The anodized aluminum was placed along the right wall of the chamber (as looking at the grating from the lamp's perspective). The spectrum that the grating produces from the incident beam not only has UV, but light in the visible spectrum as well. It is possible to extrapolate from the position of the visible spectrum the position of the UV spectrum. The anodized aluminum was placed so that part of it sat in the visible light, and the rest was where the UV should be.

The first test duration was five days. The chamber was under vacuum, and the arc lamp's beam was directed onto the grating. However, there was no color change on the aluminum, as was hoped for.

The second test was similar to the first, but with no vacuum and one other addition. A second piece was placed on top of the grating, within the arc lamp's beam. This was to see if the incident beam held enough UV to induce the desired color fade. After a three day exposure, there was still no color fading on either piece of aluminum.

Silicone S383

The first silicone test was the same as the second aluminum test, but was done in vacuum conditions. The piece of silicone on top of the grating was seen to begin fluorescing after 3.5 hours of exposure, and was fluorescing brightly after 48 hours of exposure. The test was concluded after 216 hours (nine days). At the end of the test, there was no fluorescence on the piece of silicone in the reflected path. However, there was an unexpected side effect on the piece on top of the grating. That piece is the shape of a square (roughly) with four circles cut out of it. On the back side of that piece there was also some fluorescence, apparently from the reflection off the back wall of the chamber, behind the grating and silicone.

The second silicone test performed was done with a different configuration of the grating and silicone within the chamber. Also a piece of doped polystyrene was included. The grating was set at near the middle of the chamber, at an angle of approximately 45 degrees, with respect to the incident beam. This was done to further approximate the optimal conditions for the grating.

The second test was concluded after 72 hours (3 days), when fluorescence was seen on the silicone in the reflected path. However, the chamber was brought up to air pressure, for two days, before the samples were removed and checked for fluorescence outside the chamber. For some unknown reason, there was no fluorescence on the silicone when it was removed from the chamber. No fluorescence was ever detected on the polystyrene.

The third test utilized the same configuration with the arc lamp and the grating, but several other changes were made. First, a microwave cavity lamp was attached to the chamber, in a position so that the VUV would be incident on the back side of the sample to be run, and would be otherwise blocked off from the chamber to prevent any unwanted scattering or reflections to interfere with the other test. Second, there were two strips of silicone put into the chamber, using a sample holder originally designed for the *In-Situ* facility. Also placed in the holder was a piece of aluminum foil, with cuts of equal spacing on one edge to provide an internal marking system within the chamber.

After the test was started, it was determined that the grating had been placed too low in the chamber with respect to the samples. When the incident beam had been focused onto the face of the grating, the spectrum was not on the top sample of silicone. Therefore, when fluorescence was found on the silicone, it was only on the bottom sample.

The test was stopped after 340 hours (two weeks and four hours). To prevent any reoccurence of any fading of the fluorescence due to elapsed time or atmospheric contamination, the chamber was brought to atmospheric pressure with nitrogen gas, and was transported to a workbench that had been set up beforehand in a plastic bag filled with nitrogen. After careful examination, fluorescence was determined to take place on the front right end of the bottom sample. There appeared to be two different types and areas of fluorescence on that sample. Approximately 0.09 inches from the right edge there is a section that fluoresces brightly, and starting from 1.03 inches to 0.09 inches from that edge is a section that fluoresces faintly. The faint fluorescence was unable to be seen from outside the chamber, while the bright fluorescence was highly visible from outside the chamber. Fluorescence was also found on the back center of both pieces, in the shape of ends of circles.

Problems Encountered

There were several delays and problems encountered in the preparation of these tests. The most hindering problem was that, after the second silicone test, the igniter for the arc lamp broke. Due to the age of the power supply and igniter, it could not be repaired. For the third silicone test, a power supply and igniter were borrowed from another system for the test duration. This left the system without a main radiation source.

Another potentially lengthy problem was that the system's ion pump, which is the main pump, has been temperamental. On several occasions it overheated and shut down. There have been no recent problems with it, but it is a source of concern.

The big problem on the third silicone test was the determination of the wavelengths that caused the fluorescence. The scale on the wavelengths as they landed on the sample were not linear. Apparently the sample intersected the "arc" of the spectrum at an angle that caused more wavelengths per millimeter at the right end than on the left end of the sample.

There are several ways to avoid this problem and/or to work around it. First, if the grating was situated in the exact center of the chamber, the spectrum would be spread out evenly along the inner wall of the chamber. Then, if the sample was flush against the wall, using the spectral distribution of the grating, it would be simple to determine the location of any wavelength desired. Second, several narrow band or single line filters could be placed in front of a sample while it was being irradiated. Careful measurement of the angle and distance of any fluorescence on the sample with respect to the grating would also give the information needed on wavelength.

UV Irradiation Conclusion

The UV irradiation test site is now a working configuration. When a power supply and igniter has been found for the arc lamp, the test site will have a strong UV source. The angle at which the sample intersects the spectrum needs to be closely monitored to determine what wavelengths will strike the sample. Once these problems are concluded, the system is ready for operation.

Surface Evaluation

The goal of this effort was to thoroughly characterize a variety of samples retrieved from the Long Duration Exposure Facility (LDEF). Most of the samples were thermal control type coatings or paints, while a few were metallic mirrors. A series of instruments was available within the Center for Applied Optics' Optical Metrology lab to carry out the work. The lab contained both a Wyko TOPO-3D optical profilometer and a Talystep stylus profilometer for micro-roughness and step-height measurements. There was also a Form Talysurf long-scan profilometer for measuring very rough surfaces as well as surface figure. A Zygo interferometer was also available for surface figure measurements. For scattering, a Bi-directional Reflectance Distribution Function (BRDF) instrument and a Total Integrated Scatter (TIS) instrument were available. Upon initial inspection of the samples, it was determined that the TOPO-3D should be used to measure potential steps on mirror samples. This was largely due to the ease of use of this instrument and the fact that it was non-contacting. Meanwhile, the Form Talysurf would be used to examine the rougher paint samples (they were too rough for either the TOPO-3D or the Talystep). Surface figure measurements were not required, so the Zygo was not utilized. Lastly, BRDF would be measured on the paint samples and some of the mirror samples to characterize their scattering properties. The TIS would basically be redundant with the BRDF and was not used.

Measurement Conditions/Procedures

During initial measurements of the first set of samples, the conditions of measurement on each instrument were adjusted and optimized with respect to the samples. For step-height measurements on the TOPO-3D, the expected steps were on the order of a few hundred angstroms. Since this was much less than the half-wave limit of 3300 Å on single-wavelength measurements, the multiple-wavelength option (normally used for step-heights) did not have to be utilized. The 10X objective was used (1000 x 1000 μm) to insure good coverage of the transition region. Two measurements were made on each sample on opposite sides. Pits, scratches, and other large surface features were avoided. The results were plotted in 3 dimensions as well as in 2D (perpendicular to the transition edge).

For roughness measurements on the Form Talysurf, the 60 mm length diamond stylus was used (2 μm radius tip). At first, two 5 mm long scans were made parallel to each other along the edge closest to the label (about 1/4" in from the edge). With such a short scan, which is normal for roughness measurements, one large feature (a bump or pit) could easily dominate the resulting rms roughness value. For this reason, the scan length was increased to 20 mm. This was hoped to give a better average of the surface roughness features along the nominally flat samples. Also, the second scan was changed to be perpendicular to the first along the left edge (with the label facing front). This would highlight any features with a preferred orientation. Again, extremely large features were avoided in the measurements. The resulting profile was plotted and the surface statistics printed (the rms value of the plotted data is labeled PRq on the printouts). Although the central region of the samples was avoided to preserve it from damage due to the stylus, no scratches were ever found on the samples after measurement.

The BRDF scatter measurements were made at a wavelength of 0.6328 μm. The incident beam was normal to and centered on the sample, and the scatter was measured from 5° to 60° in 5° increments (except for the last set, where the increment was reduced to 1°). A special mount was fabricated to hold the paint samples without damaging the front surface. No polarization filters were deemed necessary for these measurements. The system was periodically calibrated with a certified Lambertian reflector. In the beginning, the beam was focused at the detector (as is normal for the measurement) resulting in a spot size at the sample of about 1 mm diameter. During the first measurements, it was determined that for the paint samples the beam size should be much larger at the surface in order to average over more of the features. This would prevent one isolated bump or pit from overly influencing the results (similar to the situation above with the Form Talysurf). Thus, the beam was re-focused to give an approximately 12 mm diameter spot at the sample (for the few mirror samples, measurements were made with both a 1 mm and 12 mm spot). The results were plotted and the raw data printed out for each sample. The plots are on log-log scales with BRDF in 1/steradians versus the scatter angle in direction-cosine space notation (β-β0). BRDF is defined as the scattered radiance (watts/cm^2-ster) divided by the incident irradiance (watts/cm^2). Direction cosine space is defined as the sine of the scatter angle (β) minus the sine of the incident angle (β0). So, in this case, the values along the horizontal axis simply correspond to the sine of the scatter angle (which goes from 5° to 60°).

Measurement Results

All measurement results were delivered to NASA/MSFC as they were completed during this effort. For this reason, and due to the rather large amount of data involved, the individual plotted and/or printed results for each measurement will not accompany this report. However, a full set of these results is maintained at the CAO and can be reproduced and delivered upon request. Although a complete analysis of the results (with respect to sample composition, conditions of space exposure, etc.) must be made by the individual LDEF investigators, a few general comments can be made here.

For the partially-exposed mirror samples measured on the TOPO-3D, no discernable step was found in the transition region on any of the samples. However, in light of the fact that these samples had roughness levels between 50 and 200 Å rms, the expected step sizes of 100 to 200 Å would be almost impossible to detect. In order to accurately measure such step-heights, the sample roughness should ideally be on the order of 10 to 20 Å rms.

For the paint samples measured on the Form Talysurf, at least half showed a significant difference in the rms roughness value between the two orthogonal scans. However, upon close examination, it could be seen that these differences were generally the result of the unusually long scans. In this situation, waviness or long undulations in the surface can easily dominate the rms value while the micro-roughness levels are seen to be comparable. Such undulations could be due to the sample substrates, the way the paints were applied, or possibly due to the space exposure. The fact that about half of these samples had higher rms values on the first scan (the "A" scan) and half on the second scan (the "B" scan) suggests that this was not caused by the space exposure. Of course, this assumes that all of the samples were oriented on the LDEF platform in a like manner with respect to their labels. To summarize, there was no consistent difference between the orthogonal scans on the paint samples, and the micro-roughness levels were generally the same between them. The sample labeled "Control" was the smoothest measured at less than 1 μm rms. Most of the others were between 5 and 15 μm rms, except for samples 01-14, 01-44, and 01-54 which measured between 1 and 3 μm rms.

The paint samples measured for BRDF consistently showed a flat response at a level close to 1/pi. This indicates a very rough, Lambertian-type surface with high reflectance at the measurement wavelength. Some small

changes in the scatter at higher angles can be seen between samples (with the smoother ones dropping off a bit more rapidly). However, significant effects on the visible light scattering properties of these samples were not observed. BRDF at a longer wavelength (infrared) may be a better alternative for such samples in the future. The mirror samples showed scatter distributions consistent with their measured roughness values with no significant anomalies.