Material Qualification Test Report

Deep Sky Black – Full Spectrum Basic

External Release

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1. Scope

This document details the qualification test results for the Deep Sky Black – Full Spectrum Basic (DSB-FSB) optical black coating. Qualification testing was performed per the qualification plan where possible. Deviations from the qualification plan are documented and explained for each test sequence. References to the qualification plan, relevant material specification and supplier contact information are provided below.

Sandia M-Spec: M02050 *Anodic Coating of Aluminum to Minimize Refelctance, Deep Sky Black – Full Spectrum Basic (DSB-FSB)*

Sandia Qualification Plan: Z02050-003 *Material Qualification Plan MO2050 Anodic Coating of Aluminum to Minimize Reflectance, Deep Sky Black, Full Spectrum Basic*

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2. Qualification Test Summary

The test requirements matrix and qualification status summary are provided in Table 1. Detailed discussion for each specific test sequence are provided in subsequent sections of this document.

Test Type	Sample Size, # Failures allowed	Acceptance or Inspection Criteria	Test Result	Qualification Status
Dimensional Change	Sample Size = 3 No Failures	Shall not increase by more than 50 μm.	Average thickness change = 13.4 microns, sigma = 33.52 microns	**PASS**
Particulate Generation	Sample Size = 1 No Failures	No evidence of particulate after vibration.	No evidence of particulate after vibration	**PASS**
Local Thermal Stress	Sample Size = 1 No Failures	No visually observable change in uniformity.	Visible change in color, determined not to be of significant impact	**PASS**
Outgassing, NASA CTVS	Sample Size = 1 No Failures	Total Mass Loss ≤ 1.0%, Volatile Condensable Material ≤ 0.1% (water vapor subtracted).	TML 0.23 %, WVR corrected TML 0.08%	**PASS**
Outgassing, Residual Analysis	Sample Size = 1	Informational test only.	No outgassing components identified.	**PASS**
Optical Properties (Pre- and Post-Thermal Vacuum)	Sample Size = 3 (1 Sample Post TV) No Failures	Total hemispherical reflectance ≤ 0.25% (0.25 to 1.10 μm).	Hemispherical Reflectance < 0.02%	**PASS**

TABLE 1: TEST REQUIREMENTS MATRIX AND RESULTS SUMMARY

3. Qualification Test Results

3.1. Dimensional Change

3.1.1. Evaluation Criteria

Application of DSB-FSB coating shall not cause a dimensional change of greater than 50 μm. Evaluation was performed on three samples, with no failures allowed.

3.1.2. Test Method

Pre- and post-coating measurements were obtained using a dial indicator and flat block. Measurements were to the nearest 0.0001 inches.

3.1.3. Deviations from Qualification Plan

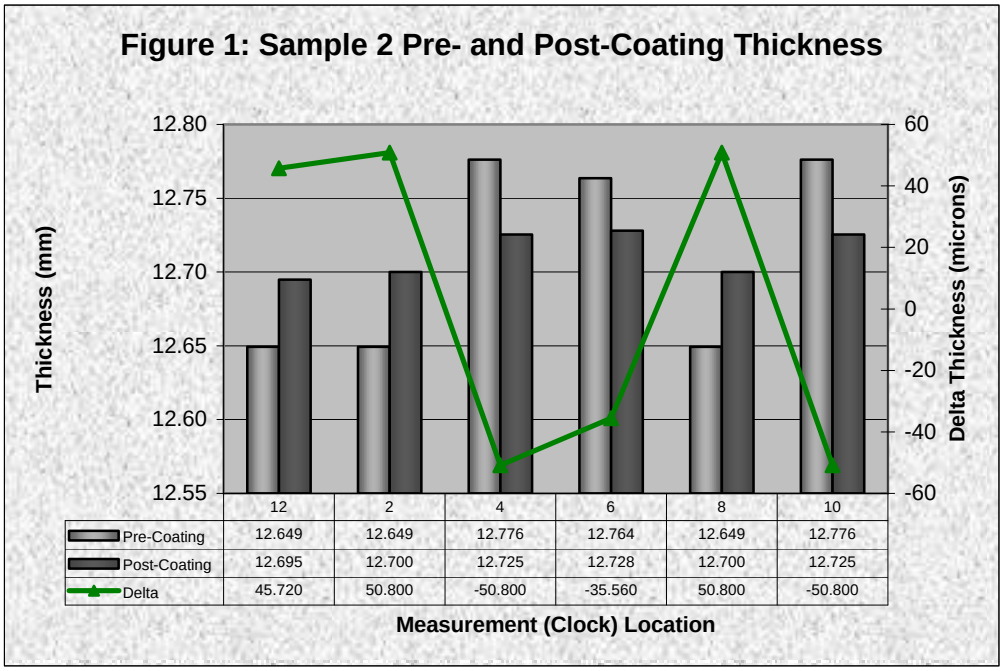
The qualification plan specified that thickness would be measured by sandwiching two samples together and measuring to the nearest 100 μ–in. The method was changed to improve test resolution for each sample. The original method confounded the results of two samples.

3.1.4. Test Results

The DSB-FSB coating passed this qualification test sequence.

Average thickness change from all samples was 13.4 microns with a standard deviation of 33.52 microns. Variability in measurements was higher than expected. Completion of a gauge repeatability and reproducibility study will be required to understand and explain the components of variability. Pre- and post-coating thickness data for each Sample is provided in Figures 1 through 3.

Figure 1: Sample 2 Pre- and Post-Coating Thickness



	12	2	4	6	8	10
Pre-Coating	12.649	12.649	12.776	12.764	12.649	12.776
Post-Coating	12.695	12.700	12.725	12.728	12.700	12.725
Delta	45.720	50.800	-50.800	-35.560	50.800	-50.800

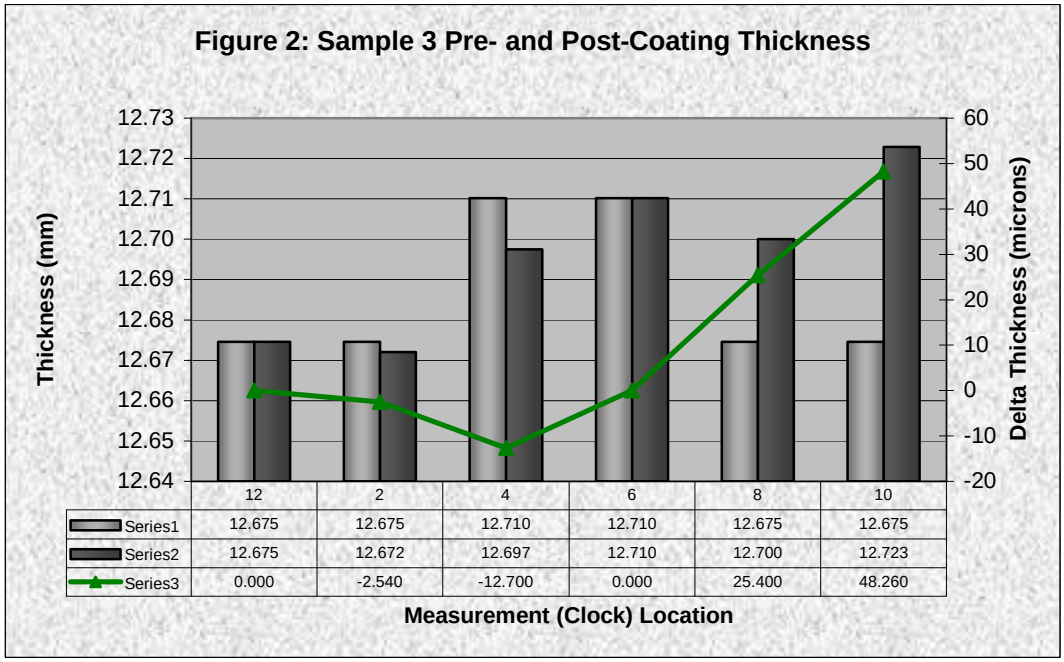


Figure 2: Sample 3 Pre- and Post-Coating Thickness
Thickness (mm)
12.73
12.72
12.71
12.70
12.69
12.68
12.67
12.66
12.65
12.64
Delta Thickness (microns)
60
50
40
30
20
10
0
-10
-20
12 2 4 6 8 10
Series1 12.675 12.675 12.710 12.710 12.675 12.675
Series2 12.675 12.672 12.697 12.710 12.700 12.723
Series3 0.000 -2.540 -12.700 0.000 25.400 48.260
Measurement (Clock) Location

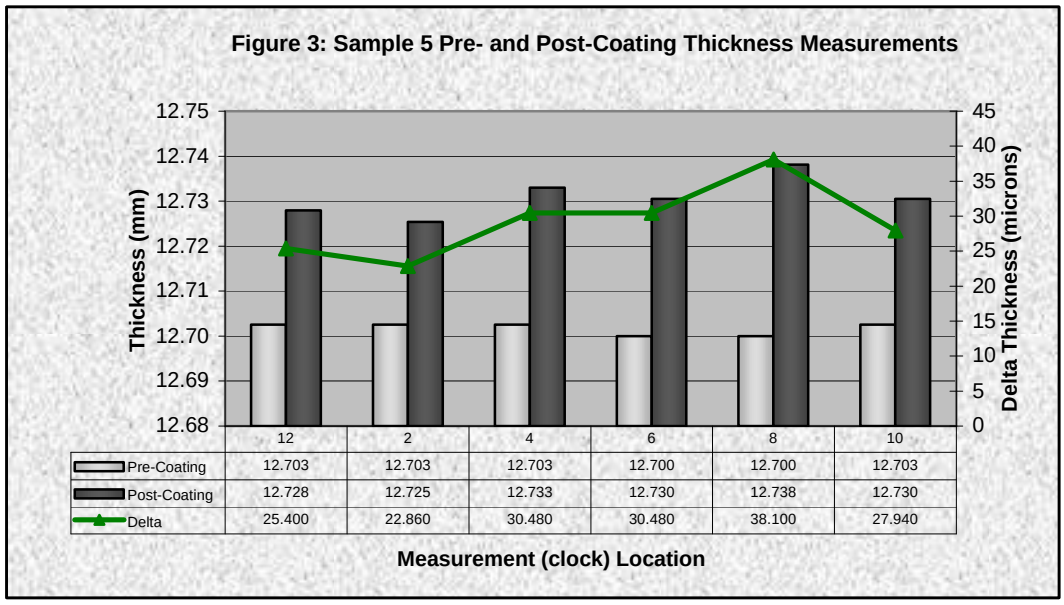


Figure 3: Sample 5 Pre- and Post-Coating Thickness Measurements
Thickness (mm)
12.75
12.74
12.73
12.72
12.71
12.70
12.69
12.68
Delta Thickness (microns)
45
40
35
30
25
20
15
10
5
0
12 2 4 6 8 10
Pre-Coating 12.703 12.703 12.703 12.700 12.700 12.703
Post-Coating 12.728 12.725 12.733 12.730 12.738 12.730
Delta 25.400 22.860 30.480 30.480 38.100 27.940
Measurement (clock) Location

3.2. Particulate Generation

3.2.1. Test Method

Assemble detector head assembly (lacking all electronics) using a DSB-FSB coated field stop. Perform thermal vacuum cycles, inspect unit for evolution of particulate. Perform random vibration at 14 Grms on each axis and inspect the unit for evolution of particulate.

3.2.2. Evaluation Criteria

DSB-FSB coating shall not generate/evolve particles before, during, or after thermal vacuum or random vibration environmental testing.

3.2.3. Deviations from Qualification Plan

A custom enclosed fixture was fabricated to hold the field stop during random vibration, the detector head assembly was not used. The enclosure was painted white to provide adequate contrast for visual examination of black-colored DSB-FSB particulate. A Pro Engineer representation of the fixture enclosure is provided in Figure 4. The DSB-FSB coated field stop is represented by the gold-colored component. Figure 5 shows the coated field stop mounted in the fixture with the top cover removed.

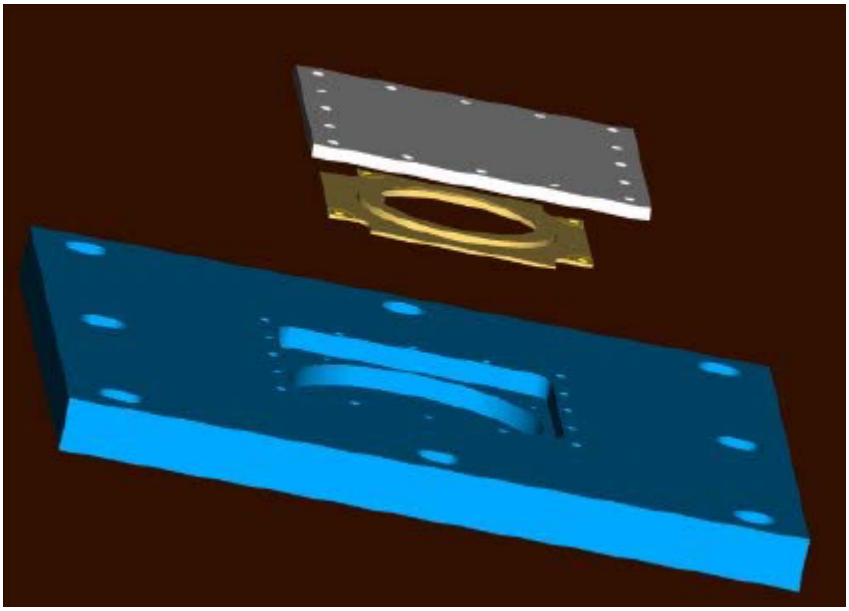


FIGURE 4: DSB-FSB VIBRATION FIXTURE

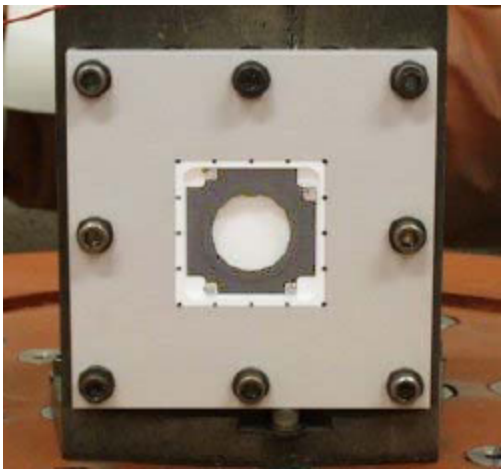


FIGURE 5: COATED FIELD STOP MOUNTED IN FIXTURE (COVER REMOVED).

3.2.4. Test Results

The DSB-FSB coating passed this qualification test sequence.

No physical coating degradation or evolution of particulate occurred during thermal vacuum or random vibration.

3.3. Local Thermal Stress

3.3.1. Test Method

Use and Argon-ion or Nd:YAG laser and appropriate focusing optics to create a circular spot of 1.3 $\pm$ 0.1 mm^2 area and 5.0 $\pm$ 0.5 W power. Ideal energy distribution should be top-hat, but significant deviations are acceptable provided beam profiles are measured and recorded. Expose DSB-FSB coating to total of 20 cycles, each having a 2-hour exposure time. The sample shall be cooled back to ambient temperature between cycles.

3.3.2. Evaluation Criteria

DSB-FSB coating shall not exhibit any visually apparent change in uniformity subsequent to local thermal stress testing.

3.3.3. Deviations from Qualification Plan

None.

3.3.4. Test Results

By strict definition, the DSB-FSB coating *did not* pass the evaluation criteria stated in 3.3.2, however, characterization of the observed changes indicate no significant degradation in the coating integrity or performance. Based on post-test characterization, the DSB-FSB coating *passed* this qualification test sequence (see discussion below).

After the first cycle, the DSB-FSB coating showed slight color change in the irradiated area. Visual examination showed a color change from deep black to a brown-black, however, the reflectivity of surface appeared to be unchanged. Both the appearance and size of this area were monitored during the remaining 19 cycles, no subsequent change in either attribute was noted.

Scanning electron microscopy was the only analysis tool able to resolve differences between the affected and unaffected areas. Due to the small affected area, quantitative reflectivity measurements could not be obtained, and optical microscopy techniques were not successful due to inadequate reflected light. SEM showed that the affected area was slightly smoother than the unaffected area, refer to Figures 6 through 8. The white spots inside the affected area (Figure 6) were of some concern, as they may indicate breaches in the coating. Additional analysis showed these areas to be crater-like features which contain a continuous layer of coating inside. This observation indicates there are regions where the coating bridges over itself, exposure to the high-energy laser beam knocked the top off creating a crater-like feature. The edges of these craters were charging which created the white spots in the image. To confirm a breach was not present, backscatter imaging was performed. Contrast in backscatter mode is produced by differences in atomic number, hence the aluminum substrate would show a significant difference in brightness compared to the DSB-FSB coating. Backscatter imaging confirmed that breaches in the coating did not exist.

Although this characterization shows no significant degradation in the coating, possible evolution of particulate was not considered in this test sequence.

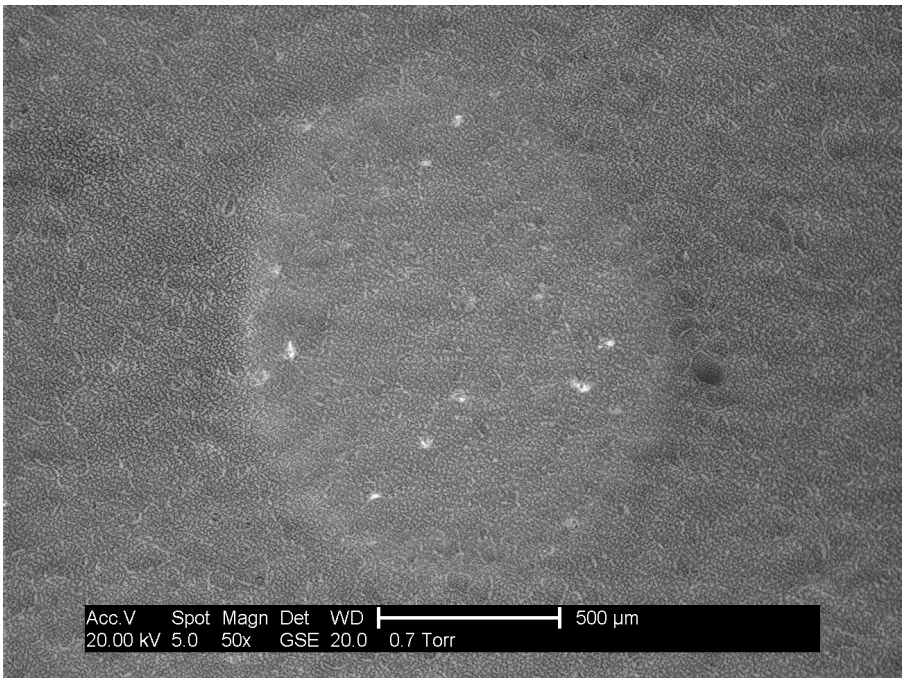
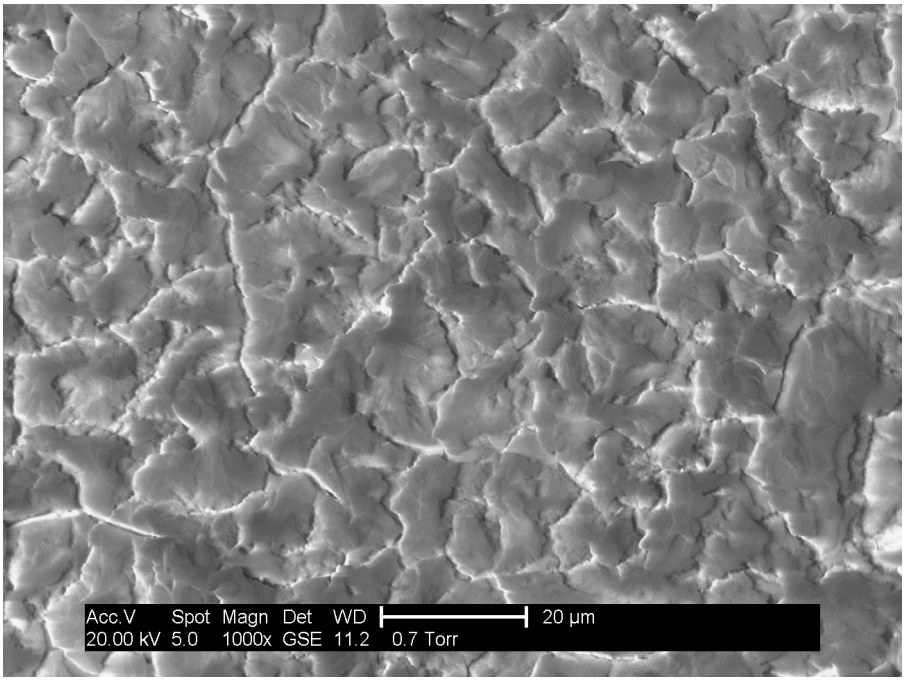
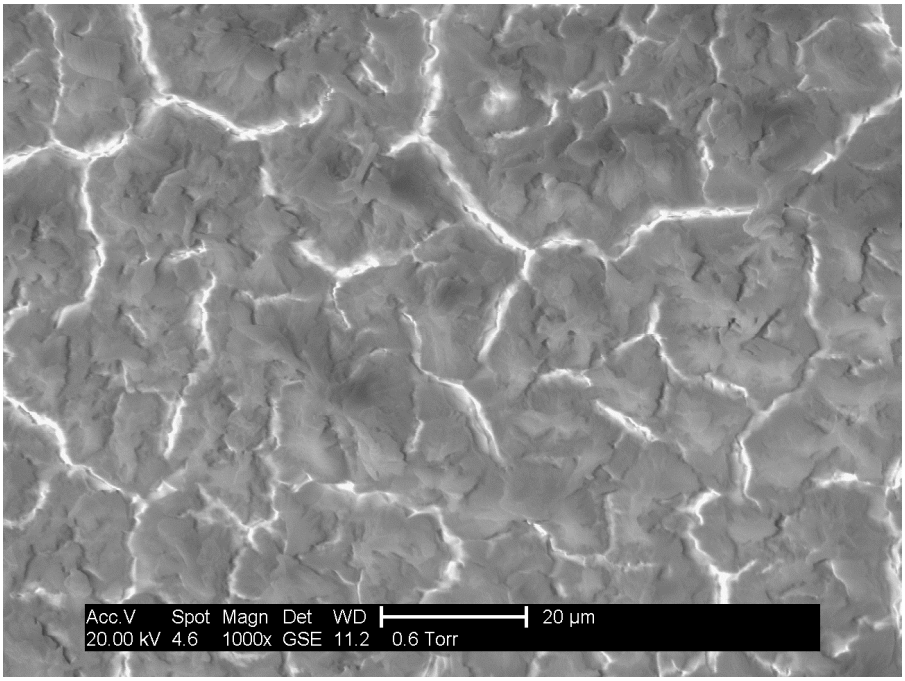


FIGURE 6: LOW MAGNIFICATION SEM MICROGRAPH OF AFFECTED/UNAFFECTED AREAS



(A)



(B)

FIGURE 7: HIGH MAGNIFICATION SEM MICROGRAPHS OF (A) UNAFFECTED AREA AND (B) AFFECTED AREA

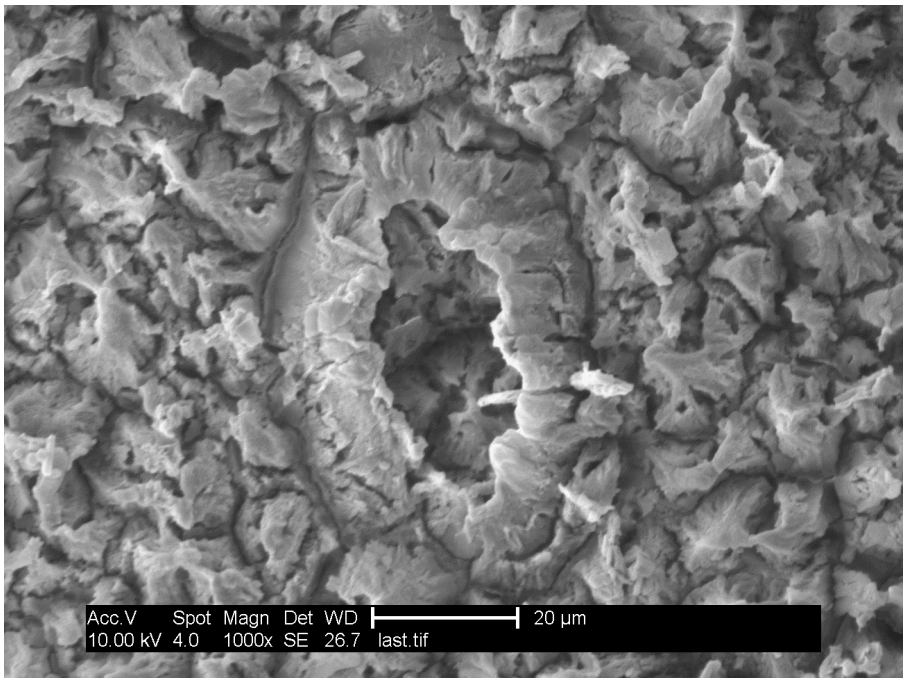


FIGURE 8: HIGH MAGNIFICATION SEM MICROGRAPH OF CRATER-LIKE FEATURE. NOTE THAT INSIDE SURFACES OF CRATER CONTAIN CONTINUOUS COATING.

3.4. Outgassing – NASA Configuration Thermal Vacuum Stability

3.4.1. Test Method

Perform ASTM E 595 outgas testing on a DSB-FSB-coated sample coupon. Analysis to include total mass loss (TML), collected volatile condensable materials (CVCM), percent water vapor recovered (WVR) and a calculation of the mass loss per unit area.

3.4.2. Evaluation Criteria

TML shall not exceed 1.00%. CVCM shall not exceed 0.10%.

3.4.3. Deviations from Qualification Plan

Analysis of the coating properties showed that ASTM E 595 was not a suitable test method for the DSB-FSB coating. The ASTM E 595 procedure dictates a coating mass between 100 and 300 mg, with a physical size limitation of approximately 1-inch (max dimension for any side). To obtain a coating mass within the specified parameters, a sample with dimensions of at least 1.5 inches per side is required.

NASA test method SP-R-0022A, *Configuration Thermal Vacuum Stability* (CTVS), was identified as a replacement for ASTM E 595. NASA developed this test method to provide a replacement for materials which would not fit into the physical guidelines of ASTM E 595. The CTVS test method is more rigorous than ASTM E 595 as it provides outgassing rates in addition to the other parameters.

3.4.4. Test Results

The DSB-FSB coating passed this qualification test sequence.

No outgassing rates or CVCM were reported during the CTVS test. All rates were below the equipment baseline rate of 5.5 X 10^{-10} g/cm^2/sec. TML, and WVR data are given in Table 1.

Parameter	Pre-Test Mass (g)	Post-Test Mass (g)	Mass (Loss) or Recovered (g)	% Change	Test Status
TML	**19.255**	**19.210**	**(0.045)**	**0.23 %**	**PASS**
WVR	**19.210**	**19.238**	**0.028**	**0.15%**	**N/A (info only)**
TML-WVR corrected	**19.255**	**19.238**	**(0.017)**	**0.08%**	**PASS**

TABLE 2: CTVS OUTGASSING TEST DATA

3.5. Outgassing – Residual Analysis

3.5.1. Test Method

Determine potential components of outgassing via FTIR analysis.

3.5.2. Evaluation Criteria

None, informational test only.

3.5.3. Deviations from Qualification Plan

None.

3.5.4. Test Results

Raman and IR spectroscopy were performed on DSB-FSB samples. Analysis shows the main component of the coating to be inorganic carbon. The analysis also showed no evidence of evolved organic material or outgassing. Based on these results the following observations and recommendations were made.

1. DSB-FSB coating will act as a getter. Care should be exercised to assure other organics are not adsorbed during integration processes. If the coating adsorbs other materials, they will likely outgas at a later time.

2. A full bake-out process followed by isolation is recommended to assure adsorbed materials are removed under controlled conditions.

3.6. Optical Properties

3.6.1. Test Method

Hemispherical Reflectance, $R_{8/D}(\lambda)$ with an incoming beam at 6 to 8 degrees.

3.6.2. Evaluation Criteria

Total Hemispherical Reflectance shall not exceed 0.25% for any wavelength in the range of 0.25 to 1.10 microns.

Total Hemispherical Reflectance shall not exceed 0.75% for any wavelength in the range 1.10 to 2.50 microns.

Solar-weighted Hemispherical Reflectance shall not exceed 0.30%.

3.6.3. Deviations from Qualification Plan

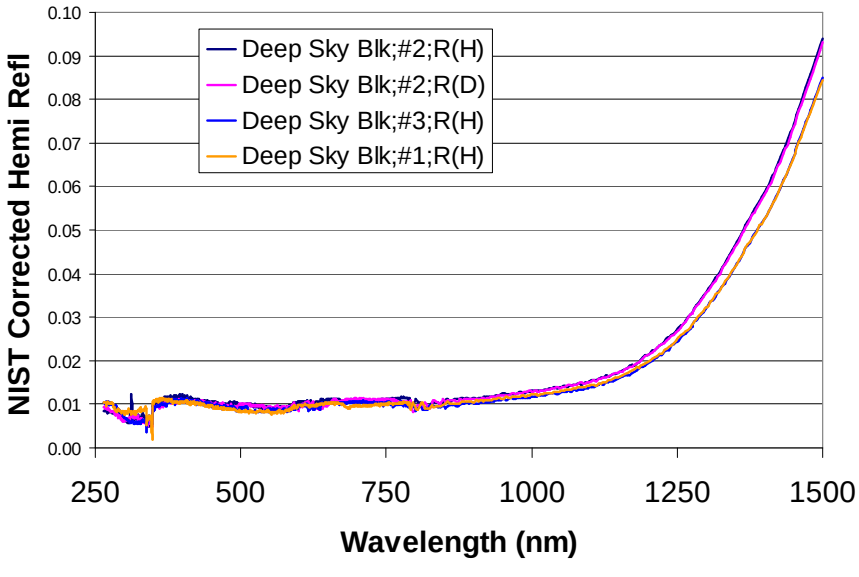
Reflectance was only measured from 0.265 to 1500 nm.

3.6.4. Test Results

The DSB-FSB coating passed this qualification test sequence.

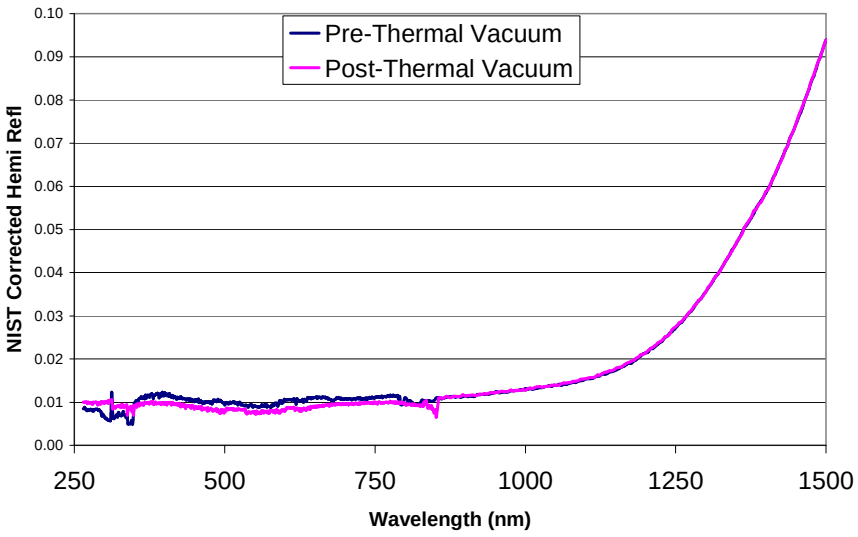
Total Hemispherical Reflectance was < 0.02% from 0.265 to 1.10 microns which is more than 12 times below the upper specification limit (refer to Figure 9). Total Hemispherical Reflectance and specular excluded measurements are nearly the same (refer to duplicate measurements for sample 2) indicating nominal lambertian reflectance characteristics for the near-normal incident angle measurement condition. The difference noted for each sample measurement is within the measurement uncertaintly of 0.5%.

Figure 9: Deep Sky Black Hemispherical Reflectance



Sample 2 was again measured subsequent to 5 thermal vacuum cycles (-40 to 80 C). Pre- and post-test data are given in Figure 10. The t-statistic for these measurements shows there is no significant difference between the pre- and post-test measurements. The t-value and confidence interval are shown in Table 3.

Figure 10: Deep Sky Black Pre- and Post-Thermal Vacuum Measurements



Wavelength Band	Pre-Test Band Average Hemispherical Reflectance (%)	Post-Test Band Average Hemispherical Reflectance (%)	t-value ($\alpha = 0.10$)	Confidence Interval ($\alpha = 0.10$)
265-400	0.0082	0.103	t = 0.982 $T_{critical}$ = 2.015 Since t<$T_{critical}$ there is no significant difference	-0.048 to 0.016
400-600	0.0098	0.0085		
600-800	0.0106	0.0096		
800-1000	0.0112	0.0113		
1000-1200	0.0157	0.0159		
1200-1500	0.0497	0.0497		

Table 3: t-statistic and confidence interval for pre- and post-thermal vacuum measurements.