Y₂O₃ Based Spray-On Cryogenic Thermal Control Coating Study

Executive Summary

The Kennedy Space Center (KSC) Applied Physics Laboratory (APL) is developing spray-on coatings to improve reflectance and decrease solar absorptance of materials used in space exploration. These cryogenic thermal control coatings (CTCC) have been applied to multiple blends of aluminum, stainless steel, silver, and multi-layer insulation (MLI). They have also been tested with and without basecoats such as the industry's standard reflective paint, AZ-93, and a commercial spray paint.

For each sample, the percent reflectance was measured and the resultant solar absorptance (SA) was calculated. Results are given in Table 1. A lower solar absorptance indicates that the CTCC-coated material will absorb less incident radiation from the sun and, therefore, will retain a lower temperature.

The best CTCC spray coating has achieved very low solar absorptance values. As a standalone coating, without any additional basecoat, a solar absorptance below 5% has been measured on 3003 aluminum, silver foil, MLI, and 7075 aluminum. Thicker coatings of CTCC spray result in better solar absorptances, to a point, and improved are possible on stainless steel. The best results measured at this time are below 3% solar absorptance.

CTCC spray has also improved the solar absorptance when applied over a basecoat of reflective paint. While AZ-93 reflective paint alone has a solar absorptance of about 15%, the addition of CTCC spray reduced solar absorptance to below 5%.

These results include solar absorptance due to water that is present on the surface of the samples in a laboratory environment. In space, this water would offgas, improving solar absorptance. Conservative estimates of removing the water absorption peaks reduce the best solar absorptance measured for any CTCC-coated samples to 2.5%.

Substrate	Basecoat/Spray	Sample	SA	Notes
3003	3 coats CTCC 9	Al-6	16.4%	
Aluminum		Al-7	13.5%	
		Al-8	18.2%	
	4 coats CTCC 10	Al-13	11.9%	Improved formulation.
	4 coats CTCC 10b	Al-14	10.0%	Ball milling improved coating.
	8 coats CTCC 10b	Al-15	9.0%	
	10 coats CTCC 10b	Al-16	7.2%	
	10 coats CTCC 10b	Al-17	4.7%	Thicker coats than Al-16.
	AZ-93 paint + 3 coats CTCC 9	Al-1	7.2%	
		Al-2	7.0%	
		Al-3	5.9%	
	AZ-93 paint + 8 coats CTCC 10b	Al-5	4.7%	
	AZ-93 paint + 10 coats CTCC 10b	Al-4	4.2%	
	White spray paint +	Al-10	10.5%	
	4 coats CTCC 10b	Al-11	10.1%	
		Al-12	10.9%	
316-2b	3 coats CTCC 9	SS-6	15.9%	
Stainless		SS-7	14.4%	
Steel		SS-8	14.6%	
	1 thick pour CTCC 9	SS-5	6.0%	Single, thick coat cracked.
	8 coats CTCC 10b	SS-15	10.4%	
	10 coats CTCC 10b	SS-14	7.2%	Similar spray thickness to Al-16.
	AZ-93 paint + 3 coats CTCC 9	SS-1	7.0%	
		SS-2	7.5%	
		SS-3	7.2%	
Silver Foil	10 coats CTCC 10b	Ag-2	4.3%	Surface not scuffed.
		Ag-3	3.3%	Surface scuffed.
Multi-Layer	10 coats CTCC 10b	MLI-1	4.4%	
Insulation		MLI-2	4.1%	
(MLI)		MLI-3	4.7%	
		MLI-4	4.9%	
7075	10 coats CTCC 10b	C-1	2.8%	Surface not scuffed.
Aluminum		C-2	3.6%	Surface scuffed.

Table 1. Summary of solar absorptance (SA) results for tested samples.

Introduction

The Solar White project has been investigating various materials that are highly reflective for use in space applications. The overall goal of the project is to find a material that will reduce solar absorption, thereby reducing the overall temperature of vehicles and vessels in space. Having a lower temperature expands the possibilities for numerous applications, including cryogenics and superconductors. Until recently, Solar White focused on fabricating rigid reflective materials.

In addition to rigid materials, however, numerous applications also exist for spray-on versions of this material, including the ability to coat cryogenics tanks and spacecraft. Several paints and coatings that are highly reflective are currently available, but their reflectivity is especially poor in the ultraviolet range of the electromagnetic (EM) spectrum.

The current, commercially available, reflective paints are expensive and are often difficult to use. They include complicated mixing and application processes. Many have lengthy curing cycles, some of which require all parts to cure in an environmental chamber, making it hard to coat large components.

Following the success of Solar White's rigid Y₂O₃ work, a liquid version was developed in the hope of achieving a product which could be equally effective or better than commercially available reflective paints, with lower cost and faster and easier processes for preparation, application, and curing. Testing has been performed to optimize the formulation and methods used, with the product being applied to aluminum, stainless steel, silver, and multi-layer insulation (MLI) in initial tests. In addition, the coating has been tested alone as well as being tested as an overspray to improve reflectivity over basecoats of AZ-93 paint and traditional commercially available spray paint.

Test Methods

To determine the effectiveness of various CTCC formulations, the CTCC formulations were applied to several substrates. CTCC formulations were also tested with and without painted basecoats. Spectral analysis was performed on the samples to compare their solar absorptions.

Spectral Analysis

Spectral analysis was performed after painting the substrates, when applicable, and after applying coats of CTCC spray. Initial data were collected using a Jasco V-670 with its ISN-723 integrating sphere accessory. After laboratory upgrades, data were collected using a Jasco V-770 with its ISN-923 integrating sphere accessory. Reflectance data were collected as percent reflectance versus wavelength from 200-2500 nm. Appendix A explains issues inherent in using this method.

All testing used Spectralon[®] standards for baseline corrections, but different Spectralon[®] samples were used with the two Jasco instruments. The older data had baseline corrections which used a Spectralon[®] sample provided by Jasco. The newer measurements were made using a NIST-traceable Spectralon[®] (SN 3153) for baseline corrections. Comparisons of data collected using each Spectralon[®] showed some difference, as the older Spectralon[®] standard had degraded or become contaminated over time. To normalize older and newer datasets to one another, a mathematical correction was applied to the older data. Appendix B presents details of this correction.

In addition to the baseline correction, a dark correction was performed on the reflectance data. Other parameters for reflectance data collected in this study were as follows:

- Data interval = 1 nm
- UV/Vis bandwidth = 2.0 nm
- NIR bandwidth = 20.0 nm
- Response time = 0.24 s
- Scan speed = 1000 nm/min

Based on the spectral data collected, the percent solar absorption for each material was calculated using ASTM E-490 data for the Sun integrated from 200 nm to 2400 nm (ASTM E903 Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres).

Substrate Selection

Four common aerospace materials were used as substrates for evaluating the spray-on coatings: aluminum panels (3003-H14 Q-panels), stainless steel panels (316-2B), silver foil, 10 layer MLI, and machined aluminum discs (7075-T651). Metal panels used were roughly 2" by 4" in size. Silver foil was roughly 1" by 1". MLI was 2" by 2", taped to aluminum panels for ease of handling and collecting reflectance measurements using the Jasco's sample holder. Aluminum discs were 1" diameter cylinders.

Metal Panel Surface Preparation

To allow for a more direct comparison between samples with and without AZ-93 paint basecoats, some metal surfaces were prepared for coating in the manner required for preparing substrates for AZ-93 paint, per the manufacturer¹:

- 1. Degrease the surface using electronics grade isopropyl alcohol (IPA) and a non-linting wipe.
- 2. Thoroughly scuff the surface using a Scotch[™]-Brite[™] pad.
- 3. Clean the surface again using electronics grade IPA and a non-linting wipe.

Figure 1 shows examples of aluminum, stainless steel, and silver substrates after surface preparation as well as a non-scuffed silver foil sample that was also used in testing.



Figure 1. Metal substrates after surface preparation. From left to right: aluminum, stainless steel, scuffed silver foil, non-scuffed silver foil.

Paint

For this study, two paints were chosen as basecoats for use with the CTCC spray. One was the aerospace industry's standard reflective paint, AZ-93 from AZ Technology. The other was a commercially available spray paint, Rust-Oleum[®] 334021 flat white spray paint.

AZ-Technologies AZ-93 Paint

AZ-93 paint was mixed following the manufacturer's guidelines²:

- 1. Add pigment and binder to a ball mill jar in specified ratios.
- 2. Add grinding media at a ratio of 1 part ³/₄" diameter to 3 parts ¹/₂" diameter, filling roughly half the jar, but not exceeding the depth of the liquid.
- 3. Roll the ball mill jar containing the mixture for 30 minutes at 70% of its critical speed.
- 4. Decant the paint from the ball mill jar by pouring it through a mesh screen.
- 5. Use within 24 hours.

Then, the Kennedy Space Center Paint Shop applied this AZ-93 to prepared aluminum and stainless steel panels, following AZ Technology recommendations¹ with minor modifications:

- 1. Pour a small amount of paint onto a wipe and rub the surface to be painted in a circular motion to prime the entire surface.
- 2. Cure prime coat for 30 minutes in 60-80% humidity at 60-95°F.
- 3. Spray an initial thin coat onto the surface using an airbrush.
- 4. Allow to dry for several minutes until it no longer appears wet.
- 5. Apply the first full coat, insuring proper wet thickness of 2-3 mil using a witness plate.
- 6. Allow to dry for several minutes until the surface is dull, rather than shiny.
- 7. Apply additional full coats until reaching the desired final wet thickness of 7-9 mil.

Once the spray coating was complete, the samples coated in AZ-93 paint were cured according to manufacturer specifications:

- 1. Day 1 and 2 60-80% humidity at 60-95°F.
- 2. Days 3 through 6 Lower humidity 5-10% per day, reaching 25-40% humidity at the end of Day 6.
- 3. Day 7 Hold at 25-40% humidity.

Spectral data were collected for the metal panels coated with an AZ-93 paint basecoat. Figure 2 shows reflectance data for AZ-93 paint on aluminum and stainless steel panels.

AZ Technology claims that this paint absorbs 14-16% of the solar spectrum³. Results were as expected, based on the manufacturer's provided information for the performance of AZ-93 paint, with calculated solar absorptions ranging from 12-17%. The measured reflectance for AZ-93 painted panels was also a close match to the vendor-provided reflectance data for their paint.



Figure 2. Reflectance of AZ-93 paint.

Rust-Oleum[®] Flat White Spray Paint

The Kennedy Space Center Paint Shop applied Rust-Oleum[®] flat white spray paint to prepared aluminum and stainless steel panels in three thicknesses: 1 mil, 2 mil, and 4 mil. The goal was to determine the minimum thickness of paint needed as a basecoat for the metal backing to no longer impact spectral reflectance measurements.

Spectral data were collected on each of the samples. Figure 3 shows the reflectance data for flat white spray paint on aluminum. Figure 4 shows the reflectance data for flat white spray paint on stainless steel.



Figure 3. Reflectance data for various thicknesses of flat white spray paint on aluminum panels.



Figure 4. Reflectance data for various thicknesses of flat white spray paint on stainless steel panels.

The spectral data showed a shift in reflectance between the 2 mil and 4 mil thick paints on both substrates. This indicated that the paint layer might not be sufficiently thick to prevent the impacts of the metal panels when collecting spectral data. As a result, an additional thick layer of spray paint was added to the aluminum panels to increase the thickness of the paint beyond the minimum amount required to block the effects of the metal in reflectance data.

Spectral data were collected again. Figure 5 shows the reflectance data for the thickest layer on all three aluminum panels compared to that collected for 4 mil thick paint on aluminum.



Figure 5. Thicker coating of white spray paint on aluminum panels.

After adding the additional coat of paint, reflectance data for all three panels were the same as that of the 4 mil thick paint, with a calculated solar absorptance of 29%. This indicated that the paint thickness was more than sufficient to prevent the metal substrate from impacting the spectral data, making them ideal for evaluating the impact of the CTCC spray coating over traditional spray paint.

CTCC Spray Coating

Several methods of applying the CTCC liquid formulations were investigated. Brushing the liquid resulted in uneven brush strokes. Application with paint rollers increases the likelihood of contaminants, as the rollers often leave small fibers behind. Pouring of the solution onto a surface resulted in too thick of a coat, which led to cracking. As such, spraying with an airbrush was chosen to be the best overall method for application.

Spray coating CTCC liquid formulations is very simple. After surface preparation and application of white or reflective paint as an undercoat, if desired, the solution is sprayed onto the surface using an airbrush. For this testing, a PointZero Single-Action 22cc Siphon-Fed Airbrush equipped with a 0.8 mm nozzle (shown in Figure 6) was used. This type of airbrush pushes gas through the nozzle, creating a siphoning effect that pulls the liquid up into the gas stream and spraying it out the nozzle. Dry nitrogen gas was used, rather than air, to insure that no additional water was added during spraying. Other styles of airbrush would likely work equally well, but have not been evaluated.



Figure 6. PointZero Single-Action 22cc Siphon-Fed Airbrush equipped with a 0.8 mm nozzle, used for application of CTCC spray-on coatings.

Spray methods have yet to be optimized. For this coating study, most CTCC spray was added to samples that were positioned horizontally. The airbrush was pointed down, forming an angle of about sixty degrees above horizontal, held at a distance of approximately six inches from the surface. This spray setup is shown in Figure 7. Spray was applied moving the airbrush slowly across the surface.



Figure 7. General alignment for horizontal CTCC spray application (not to scale).

The CTCC spray added to the MLI samples was applied while the samples were positioned vertically. In this configuration, shown in Figure 8, the spray is directed perpendicularly at the sample, rather than at an angle. In the case of MLI, this helped to prevent delamination of the coating due to the rippling motion

of the MLI during spraying. This method has been demonstrated on an aluminum panels and could be used on other materials as well.



Figure 8. General alignment for vertical CTCC spray application (not to scale).

For each coat of CTCC spray, spray was applied to the substrate as the airbrush moved across the surface left-to-right and also top-to-bottom in an attempt to achieve a more even coating. CTCC spray performs best when it is applied in several, thinner coats, and allowed to dry between each coat, rather than applying a thicker coat. This results in a more even coating and eliminates cracking while drying. The exact thickness per coat and number of coats to apply require further optimization, but current results have been good with ~0.1-0.3 g of spray added to a $2^{"} \times 4^{"}$ panel per coat.

All CTCC spray formulations were allowed to air dry in an area without strong air currents. A thin coat of spray would dry in under two hours. To speed up the overall process, it is possible that subsequent coats could be applied much sooner, but further study is required.

After the application of each coat of spray, sample masses were determined, and photos and spectral data were collected before the next coat of spray was added.

Data and Results

Two formulations of CTCC spray coat were evaluated. CTCC Formulation 9 was evaluated on aluminum and stainless steel substrates, sprayed onto both bare metal and metal with an AZ-93 paint basecoat. CTCC Formulations 10 and 10b were compared on aluminum panels. CTCC Formulation 10b was then evaluated on substrates of aluminum, stainless steel, silver foil, and MLI. In addition, it was evaluated on aluminum and stainless steel panels with a basecoat of AZ-93 paint and on aluminum panels with a basecoat of white spray paint. Results of each were compared to determine the best methods and formulations for future use.

CTCC Formulation 9

Initial spectral analysis of Y_2O_3 spray coating on metal examined a formulation of the spray on solution referred to as CTCC Formulation 9. CTCC Formulation 9 utilizes a co-solvent system composed of ultrapure water (Solvent A) and ethanol (Solvent B). The optimal formulation for the Solvent A + Solvent B

(ethanol) system is 17.3%, 13.8%, 51.7%, 17.2% (Binder, Scatterer, Solvent A, Solvent B). All percentages are Wt:Wt percent.

CTCC Formulation 9 was sprayed onto bare aluminum and stainless steel panels: Al-6, Al-7, Al-8, SS-6, SS-7, and SS-8. This formulation was also sprayed onto aluminum and stainless steel panels that had AZ-93 paint basecoats: Al-1, Al-2, Al-3, SS-1, SS-2, and SS-3. Figure 9 through Figure 20 show the metal panels spray coated with CTCC Formulation 9 after the application of each coat of spray.

Due to automatic adjustments made by the camera when photographing very reflective coatings, the pictures of AZ-93 paint and CTCC sprays do not appear as bright white as they do in person.



Figure 9. Aluminum panel Al-6 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 10. Aluminum panel Al-7 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 11. Aluminum panel Al-8 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 12. Stainless steel panel SS-6 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 13. Stainless steel panel SS-7 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 14. Stainless steel panel SS-8 sprayed with 1, 2, and 3 coats of CTCC Formulation 9.



Figure 15. Aluminum panel Al-1 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.



Figure 16. Aluminum panel Al-2 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.



Figure 17. Aluminum panel Al-3 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.



Figure 18. Stainless steel panel SS-1 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.



Figure 19. Stainless steel panel SS-2 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.



Figure 20. Stainless steel panel SS-3 with an AZ-93 paint basecoat followed by 1, 2, and 3 coats of CTCC Formulation 9 spray.

The first coat of CTCC Formulation 9 spray did not result in complete coverage of the surface. This is most readily apparent in the samples where the spray coat was applied to bare metal, but was also true for the samples with an AZ-93 paint basecoat. While coverage did improve with each coat of spray, the substrate was still visible through the spray after three coats.

After drying, CTCC Formulation 9 had a gritty texture. This was true for all substrates, but is more evident in the pictures of the spray coating over AZ-93 paint basecoats. Another challenge with CTCC Formulation 9 was that it frequently clogged the airbrush nozzle during spraying.

Figure 21 through Figure 32 show the spectral results for CTCC Formulation 9 sprayed onto aluminum and stainless steel. It is obvious that the reflectance improves with increased coverage of the CTCC Formulation 9 spray as more coats were applied. This was particularly noticeable in the ultraviolet range of the spectrum. In the samples with AZ-93 paint basecoats, the addition of CTCC spray did not result in much improvement in the longer wavelengths, but the improvement below 400 nm, where AZ-93 paint absorbs light, was striking, even after one coat of spray.



Figure 21. Reflectance data for CTCC Formulation 9 sprayed onto Al-6.



Figure 22. Reflectance data for CTCC Formulation 9 sprayed onto Al-7.



Figure 23. Reflectance data for CTCC Formulation 9 sprayed onto Al-8.



Figure 24. Reflectance data for CTCC Formulation 9 sprayed onto SS-6.



Figure 25. Reflectance data for CTCC Formulation 9 sprayed onto SS-7.



Figure 26. Reflectance data for CTCC Formulation 9 sprayed onto SS-8.



Figure 27. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on Al-1.



Figure 28. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on Al-2.



Figure 29. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on Al-3.



Figure 30. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on SS-1.



Figure 31. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on SS-2.



Figure 32. Reflectance data for CTCC Formulation 9 sprayed over AZ-93 paint on SS-3.

CTCC Formulation 9 was also poured directly onto a stainless steel panel that had an AZ-93 paint basecoat: SS-5. To do so, a mold-like structure was constructed around the panel's edges using painter's tape, and then several full droppers of CTCC Formulation 9 were dripped onto the surface.

Figure 33 shows the results. With such a thick coat of CTCC applied all at once – more than twice the total thickness of three coats of CTCC Formulation 9 sprayed onto the previous samples – the surface cracked significantly while drying. The cracked areas delaminated and flaked off during handling, leaving areas of exposed AZ-93 paint.



Figure 33. Stainless steel panel SS-5 with CTCC Formulation 9 poured in a thick coat over AZ-93 paint.

Figure 34 shows the spectral results for CTCC Formulation 9 poured onto stainless steel that had an AZ-93 paint basecoat. Like the sprayed samples, reflectance greatly improved over that of AZ-93 paint in the ultraviolet range of the spectrum. Reflectance was also better than the samples with three coats of CTCC Formulation 9 sprayed onto them, likely due to the much thicker coat of Y_2O_3 .



Figure 34. Reflectance data for CTCC Formulation 9 poured over an AZ-93 paint basecoat on a stainless steel panel.

Table 2 lists the mass of each sample (including substrate) per coat and each coat's calculated solar absorption for all of the CTCC Formulation 9 samples. The samples which had a basecoat of AZ-93 paint had much lower absorptions overall than those in which the CTCC Formulation 9 was sprayed onto bare metal. Nonetheless, the CTCC spray onto bare metal was able to reach absorptions similar to that of AZ-93 paint, the current industry standard, after only a few coats. All samples, with and without AZ-93 paint basecoats, showed marked improvement after a single coat of CTCC Formulation 9 spray, in spite of incomplete surface coverage.

The thick poured coat of CTCC Formulation 9 on SS-5 yielded the best solar absorption results. This indicates that a thicker coat of spray would result in improved results for the other samples as well, if it could be achieved without the cracking and flaking that resulted from that very thick coat.

Sample	Coating	Total Coating Mass (g)	CTCC Mass (g)	Solar Absorption
Al-6	1 coat CTCC 9	0.207	0.207	29.2%
	2 coats CTCC 9	0.356	0.356	19.9%
	3 coats CTCC 9	0.653	0.653	16.4%
Al-7	1 coat CTCC 9	0.275	0.275	25.8%
	2 coats CTCC 9	0.484	0.484	15.5%
	3 coats CTCC 9	0.772	0.772	13.5%
Al-8	1 coat CTCC 9	0.177	0.177	30.0%
	2 coats CTCC 9	0.295	0.295	25.4%
	3 coats CTCC 9	0.529	0.529	18.2%
SS-6	1 coat CTCC 9	0.206	0.206	36.0%
	2 coats CTCC 9	0.458	0.458	19.8%
	3 coats CTCC 9	0.757	0.757	15.9%
SS-7	1 coat CTCC 9	0.198	0.198	32.8%
	2 coats CTCC 9	0.404	0.404	21.1%
	3 coats CTCC 9	0.728	0.728	14.4%
SS-8	1 coat CTCC 9	0.236	0.236	27.6%
	2 coats CTCC 9	0.447	0.447	18.1%
	3 coats CTCC 9	0.771	0.771	14.6%
Al-1	AZ-93	0.570	0.000	17.0%
	AZ-93 + 1 coat CTCC 9	0.799	0.229	10.2%
	AZ-93 + 2 coats CTCC 9	0.975	0.405	8.3%
	AZ-93 + 3 coats CTCC 9	1.157	0.587	7.2%
Al-2	AZ-93	0.570	0.000	14.4%
	AZ-93 + 1 coat CTCC 9	0.818	0.248	10.7%
	AZ-93 + 2 coats CTCC 9	1.109	0.539	7.9%
	AZ-93 + 3 coats CTCC 9	1.266	0.696	7.0%
Al-3	AZ-93	0.570	0.000	14.2%
	AZ-93 + 1 coat CTCC 9	0.880	0.310	11.1%
	AZ-93 + 2 coats CTCC 9	1.134	0.564	7.1%
	AZ-93 + 3 coats CTCC 9	1.433	0.863	5.9%
SS-1	AZ-93	0.570	0.000	13.8%
	AZ-93 + 1 coat CTCC 9	0.839	0.269	9.4%
	AZ-93 + 2 coats CTCC 9	1.116	0.421	7.5%
	AZ-93 + 3 coats CTCC 9	1.458	0.888	7.0%
SS-2	AZ-93	0.570	0.000	13.7%
	AZ-93 + 1 coat CTCC 9	0.766	0.196	12.1%
	AZ-93 + 2 coats CTCC 9	0.995	0.425	8.3%
	AZ-93 + 3 coats CTCC 9	1.359	0.789	7.5%
SS-3	AZ-93	0.570	0.000	13.3%
	AZ-93 + 1 coat CTCC 9	0.691	0.121	9.5%
	AZ-93 + 2 coats CTCC 9	0.991	0.546	8.1%
	AZ-93 + 3 coats CTCC 9	1.257	0.687	7.2%
SS-5	1 coat CTCC 9 (thick pour)	2.266	1.696	6.0%

Table 2. CTCC Formulation 9 on Aluminum and Stainless Steel Substrates, with and without AZ-93 paint.

CTCC Formulations 10 and 10b

In an attempt to improve upon CTCC Formulation 9's ability to be sprayed as well as its reflectivity, a new formulation was developed, CTCC Formulation 10. CTCC Formulation 10 utilizes a co-solvent system composed of ultra-pure water (Solvent A) and isopropanol (Solvent B). The optimal formulation for the Solvent A + Solvent B (isopropanol) system is 16.1%, 19.5%, 48.3%, 16.1% (Binder, Scatterer, Solvent A, Solvent B). All percentages are Wt:Wt percent.

After obtaining the results of initial test sprays with coating CTCC Formulation 10, this new formulation was ball milled to try to break up the clumps of Y_2O_3 and achieve a more even distribution of reflecting particles in each coat of spray. A portion of the CTCC Formulation 10 solution was decanted into a second Nalgene bottle, and several pieces of 0.50" cylindrical premium yttria-stabilized zirconia ball mill media were added. It was rolled it at 30-50% speed on a U.S. Stoneware Benchtop Jar Mill. The resultant spray appeared to be much more even. To differentiate them, this version was called CTCC Formulation 10b.

Comparing CTCC Formulation 10 and CTCC Formulation 10b

An initial comparison was performed, spraying CTCC Formulation 10 onto an aluminum panel, Al-13, and spraying CTCC Formulation 10b onto another aluminum panel, Al-14. Efforts were made to keep spray volumes the same for each coat between the two samples.

As anticipated, CTCC Formulations 10 and 10b both sprayed much more easily, with almost no clogging.

Figure 35 shows four coats of CTCC Formulation 10 sprayed onto aluminum. Figure 36 shows four coats of CTCC Formulation 10b sprayed onto aluminum. Both versions sprayed much more easily and coated more evenly than CTCC Formulation 9 had. By the fourth coat, the metal backing was barely visible through the spray. Both formulations were less grainy in texture than CTCC Formulation 9 had been, but CTCC Formulation 10b, which had been ball milled, was noticeably smoother.



Figure 35. Aluminum panel Al-13 with 1, 2, 3, and 4 coats of CTCC Formulation 10 spray.



Figure 36. Aluminum panel Al-14 with 1, 2, 3, and 4 coats of CTCC Formulation 10b spray.

Figure 37 and Figure 38 show the spectral results for samples spray coated with CTCC Formulation 10 and 10b. As with CTCC Formulation 9, this new formulation of spray shows improvement with increased number of coats, although it showed little change between coats three and four on either sample. Results were similar for the regular and ball milled versions of CTCC Formulation 10, but with slightly better results on the ball milled coatings.



Figure 37. Reflectance data for CTCC Formulation 10 spray over bare aluminum, Al-13.



Figure 38. Reflectance data for CTCC Formulation 10b (ball milled) spray over bare aluminum, Al-14.

Table 3 lists the resultant solar absorption for each coat, along with the sample mass following each spray coat. CTCC Formulation 10b achieved a lower solar absorption after four coats of spray than CTCC Formulation 10. More notably, after three coats of spray, both preparations of CTCC Formulation 10 spray had lower solar absorptions than CTCC Formulation 9, showing a marked improvement with this new formulation.

Sample Al-14 had a higher solar absorption measured for its fourth coat than its third. In later work, it was determined that, due to the unevenness of the coating, particularly with fewer total coats, results can differ from one area to another. It is likely that, if additional measurements were taken at several different positions on the sample, some portions of the sample would have shown a continued improvement, rather than degradation.

Sample	Coating	CTCC Mass (g)	Solar Absorption
Al-13	None	0.000	32.3%
	1 coat CTCC 10	0.168	24.7%
	2 coats CTCC 10	0.317	19.4%
P	3 coats CTCC 10	0.581	12.0%
	4 coats CTCC 10	0.660	11.9%
Al-14	None	0.000	31.8%
	1 coat CTCC 10b	0.177	23.3%
	2 coats CTCC 10b	0.362	15.4%
	3 coats CTCC 10b	0.723	9.7%
P	4 coats CTCC 10b	0.894	10.0%

Table 3. CTCC Formulation 10 and 10b on Aluminum Panels.

CTCC Formulation 10b over White Spray Paint

With the ball-milled version of CTCC Formulation 10 showing better performance, it was selected for testing the effectiveness of spraying over a traditional flat white spray paint basecoat. The aluminum panels painted with Rust-Oleum[®] spray paint previously discussed (Al-10, Al-11, and Al-12) were chosen. The stainless steel panels with Rust-Oleum[®] paint were not used in case they were needed for testing with a future formulation of CTCC spray.

Figure 39 though Figure 41 show the metal panels painted with spray paint, then sprayed with CTCC Formulation 10b after the application of each coat of CTCC spray. CTCC Formulation 10b coated the spray painted surfaces similarly to its performance on bare aluminum, with a fairly even spray that resulted in good coverage after four coats.



Figure 39. Aluminum panel Al-10 with a flat white spray paint basecoat followed by 1, 2, 3, and 4 coats of CTCC Formulation 10b spray.



Figure 40. Aluminum panel Al-11 with a flat white spray paint basecoat followed by 1, 2, 3, and 4 coats of CTCC Formulation 10b spray.



Figure 41. Aluminum panel Al-12 with a flat white spray paint basecoat followed by 1, 2, 3, and 4 coats of CTCC Formulation 10b spray.

Figure 42 through Figure 44 show the spectral results for samples spray coated with CTCC Formulation 10b. The flat white spray paint absorbs significantly more EM radiation overall than the AZ-93 paint, as expected. As with the AZ-93 paint, the addition of even a single coat of Y_2O_3 spray yields significant improvement, particularly in the ultraviolet range. The flat white spray paint performed worse in the longer wavelengths than the AZ-93 paint, however, those wavelengths show a greater increase in performance with the addition of Y_2O_3 spray than was seen with spraying over AZ-93 paint. Unfortunately, the absorption of the flat white spray paint caused worse results overall.



Figure 42. Reflectance data for CTCC Formulation 10b spray over a flat white spray paint basecoat on sample Al-10.



Figure 43. Reflectance data for CTCC Formulation 10b spray over a flat white spray paint basecoat on sample Al-11.



Figure 44. Reflectance data for CTCC Formulation 10b spray over a flat white spray paint basecoat on sample Al-12.

Table 4 lists the resultant solar absorption for each coat, along with the sample mass following each spray coat. When comparing the solar absorptions of CTCC Formulation 10b sprayed onto uncoated aluminum to those sprayed onto aluminum with a flat white spray paint basecoat, the results after four coats were similar. After only one coat, the solar absorption was lower for the spray painted panels. This suggests that, in cases where only a small amount of Y_2O_3 spray would be applied, any white paint as a basecoat is preferential to bare metal. Still, if several coats of Y_2O_3 spray are able to be applied to achieve complete coverage, it is better not to have the traditional white spray paint basecoat, as this adds mass without improving absorption.

Sample	Coating	Total Coating Mass (g)	CTCC Mass (g)	Solar Absorption
Al-10	None	0.000	0.000	30.1%
	Flat white spray paint	1.424	0.000	29.0%
	Paint + 1 coat CTCC 10b	1.585	0.161	18.4%
	Paint + 2 coats CTCC 10b	1.799	0.375	12.7%
	Paint + 3 coats CTCC 10b	2.003	0.579	9.9%
	Paint + 4 coats CTCC 10b	2.144	0.720	10.5%
Al-11	None	0.000	0.000	32.3%
	Flat white spray paint	2.114	0.000	29.0%
	Paint + 1 coat CTCC 10b	2.225	0.111	19.2%
	Paint + 2 coats CTCC 10b	2.490	0.376	11.0%
	Paint + 3 coats CTCC 10b	2.695	0.581	9.4%
	Paint + 4 coats CTCC 10b	2.808	0.694	10.1%
Al-12	None	0.000	0.000	33.7%
	Flat white spray paint	2.852	0.000	29.0%
	Paint + 1 coat CTCC 10b	2.921	0.069	20.4%
	Paint + 2 coats CTCC 10b	3.121	0.269	12.5%
	Paint + 3 coats CTCC 10b	3.275	0.423	11.5%
	Paint + 4 coats CTCC 10b	3.377	0.525	10.9%

Table 4. CTCC Formulation 10b on Aluminum with a Rust-Oleum[®] Flat White Spray Paint basecoat.

CTCC Formulation 10b on Metal Panels with and without AZ-93 Paint

A new batch of CTCC Formulation 10b was prepared to test this formulation on an aluminum panel (Al-15), a stainless steel panel (SS-15), and an aluminum panel with an AZ-93 paint basecoat (Al-5).

Figure 45 through Figure 47 show these metal panels, sprayed with CTCC Formulation 10b, after the application of each coat of spray. These spray coats had grainier textures than those produced by the previous batch of CTCC Formulation 10b, but still sprayed well and were able to achieve thicker overall coatings than had been reached in previous testing, without cracking or flaking.



Figure 45. Aluminum panel Al-15 with 1-8 coats of CTCC Formulation 10b spray.



Figure 46. Stainless steel panel SS-15 with 1-8 coats of CTCC Formulation 10b spray.



Figure 47. Aluminum panel AI-5 with 1-8 coats of CTCC Formulation 10b spray over AZ-93 paint.

Figure 48 through Figure 50 show the spectral results for samples spray coated with CTCC Formulation 10b. As with CTCC Formulation 9, the results in the longer wavelengths were better for the sample that had an AZ-93 paint basecoat than they were for the samples on bare metal, but in the ultraviolet range, initial results were better for CTCC Formulation 10b sprayed over bare metal than for CTCC Formulation 10b sprayed over a basecoat of AZ-93 paint. While improvement per coat does decrease as the total number of coats increases, there was some improvement with each of the eight coats applied.



Figure 48. Reflectance data for Al-15 sprayed with CTCC Formulation 10b.


Figure 49. Reflectance data for SS-15 sprayed with CTCC Formulation 10b.



Figure 50. Reflectance data for AI-5 with an AZ-93 paint basecoat sprayed with CTCC Formulation 10b.

Table 5 lists resultant solar absorption for each coat, along with the sample mass following each spray coat. Comparing these results to those measured for CTCC Formulation 9 for the same substrates, CTCC Formulation 10b shows lower solar absorption for an equivalent mass of CTCC spray added. This indicates improved reflectivity for CTCC Formulation 10b over CTCC Formulation 9, in addition to the improvements in the ability to be sprayed.

Evaluating the performance of CTCC Formulation 10b on each of the substrates tested, uncoated aluminum and stainless steel were similar, with aluminum performing slightly better in the first coats due to its lower initial solar absorption. The aluminum panel with an AZ-93 paint basecoat still showed a significantly better result than the others.

Solar absorption does continue to decrease with each added coat. Testing of this set of samples stopped after eight coats so that evaluation of new samples could begin with additional ball milling; however, additional coats could have been added to achieve continued improvement for these samples.

Sample	Coating	Total Coating Mass (g)	CTCC Mass (g)	Solar Absorption
Al-15	None	0.000	0.000	31.3%
	2 coats CTCC 10b	0.240	0.240	21.1%
	3 coats CTCC 10b	0.383	0.383	19.4%
	4 coats CTCC 10b	0.572	0.572	14.8%
	5 coats CTCC 10b	0.753	0.753	10.5%
	6 coats CTCC 10b	0.945	0.945	9.5%
	7 coats CTCC 10b	1.061	1.061	9.3%
	8 coats CTCC 10b	1.182	1.182	9.0%
SS-15	None	0.000	0.000	49.8%
	2 coats CTCC 10b	0.261	0.261	26.7%
	3 coats CTCC 10b	0.424	0.424	21.1%
	4 coats CTCC 10b	0.645	0.645	14.5%
	5 coats CTCC 10b	0.867	0.867	13.5%
	6 coats CTCC 10b	1.008	1.008	11.3%
	7 coats CTCC 10b	1.082	1.082	11.5%
	8 coats CTCC 10b	1.195	1.195	10.4%
Al-5	None	0.000	0.000	~30-35%
	AZ-93 Paint	0.599	0.000	14.8%
	AZ-93 + 2 coats CTCC 10b	0.855	0.256	6.9%
	AZ-93 + 3 coats CTCC 10b	0.995	0.396	6.3%
	AZ-93 + 4 coats CTCC 10b	1.176	0.577	5.7%
	AZ-93 + 5 coats CTCC 10b	1.296	0.697	5.5%
	AZ-93 + 6 coats CTCC 10b	1.473	0.874	5.3%
	AZ-93 + 7 coats CTCC 10b	1.651	1.052	5.1%
	AZ-93 + 8 coats CTCC 10b	1.792	1.193	4.7%

Table 5. CTCC Formulation 10b on Metal Panels, with and without AZ-93 Paint.

The spray in this set of panels was clumpier than desired, which appeared to be the result of mechanical issues that occurred during the initial ball milling for the new batch of CTCC Formulation 10b that was mixed for coating samples Al-15, SS-15, and Al-5. To resolve this, the remaining formulation was ball-

milled again, and the test was repeated with a new set of metal panels: Al-16, SS-14, and Al-4 (which had an AZ-93 paint basecoat).

Figure 51 through Figure 53 show these panels, sprayed with the new batch of CTCC Formulation 10b following additional ball milling, after the application of each coat of spray. This set of samples had noticeably smoother results than AI-5, AI-15, and SS-15 had, indicating that the additional round of ball milling was a success.

For these samples, ten total coats of CTCC Formulation 10b were added before testing stopped, which was the greatest number of coats on any samples thus far. There was no delamination or cracking damage to the surface coating. This means that further improvements to reflectance and solar absorption are still possible using CTCC Formulation 10b, as well as with future, more optimized formulations.



Figure 51. Aluminum panel Al-16 with 1-10 coats of CTCC Formulation 10b spray.



Figure 52. Stainless steel panel SS-14 with 1-10 coats of CTCC Formulation 10b spray.



Figure 53. Aluminum panel Al-4 with 1-10 coats of CTCC Formulation 10b spray over AZ-93 paint.

Figure 54 through Figure 56 show the spectral results for samples spray coated with CTCC Formulation 10b. Results were similar to those for Al-15, SS-15, and Al-5, but with some improvement seen by the eighth coat applied. After the tenth coat, these samples had the best results seen so far.



Figure 54. Reflectance data for Al-16 sprayed with CTCC Formulation 10b.



Figure 55. Reflectance data for SS-14 sprayed with CTCC Formulation 10b.



Figure 56. Reflectance data for Al-4 with CTCC Formulation 10b sprayed over AZ-93 paint.

Table 6 lists resultant solar absorptions for each coat along with the sample mass following each spray coat. In spite of starting out with much better results on aluminum than stainless steel, these two substrates had identical solar absorptions after the tenth coat. This could be due to the Y_2O_3 layer being thick enough and having full enough coverage, at that point, to prevent the metal backing from having an impact on the overall result.

While additional coats could be added to further improve reflectance, after ten coats of CTCC Formulation 10b on metal substrates with no basecoat, the measured solar absorption was half of that measured for AZ-93 paint alone. With AZ-93 paint and CTCC Formulation 10b working together, the solar absorption was reduced to less than one-third of the solar absorption for AZ-93 paint alone.

Sample	Coating	Total Coating Mass (g)	CTCC Mass (g)	Solar Absorption
Al-16	None	0.000	0.000	29.5%
	1 coat CTCC 10b	0.098	0.098	27.6%
	2 coats CTCC 10b	0.189	0.189	23.9%
	3 coats CTCC 10b	0.310	0.310	18.2%
	4 coats CTCC 10b	0.534	0.534	13.8%
	5 coats CTCC 10b	0.786	0.786	12.0%
	6 coats CTCC 10b	0.874	0.874	10.5%
	7 coats CTCC 10b	1.023	1.023	10.4%
	8 coats CTCC 10b	1.330	1.330	7.8%
	9 coats CTCC 10b	1.444	1.444	7.6%
	10 coats CTCC 10b	1.611	1.611	7.2%
SS-14	None	0.000	0.000	51.7%
	1 coat CTCC 10b	0.115	0.115	37.1%
	2 coats CTCC 10b	0.218	0.218	28.6%
	3 coats CTCC 10b	0.346	0.346	24.3%
	4 coats CTCC 10b	0.587	0.587	16.5%
	5 coats CTCC 10b	0.899	0.899	12.3%
	6 coats CTCC 10b	1.079	1.079	10.6%
	7 coats CTCC 10b	1.241	1.241	9.7%
	8 coats CTCC 10b	1.524	1.524	9.5%
	9 coats CTCC 10b	1.657	1.657	8.8%
	10 coats CTCC 10b	1.868	1.868	7.2%
Al-4	None	0.000	0.000	~30-35%
	AZ-93 Paint	0.570	0.000	13.9%
	AZ-93 + 1 coat CTCC 10b	0.677	0.107	10.3%
	AZ-93 + 2 coats CTCC 10b	0.777	0.207	8.0%
	AZ-93 + 3 coats CTCC 10b	0.920	0.350	7.2%
	AZ-93 + 4 coats CTCC 10b	1.034	0.464	6.6%
	AZ-93 + 5 coats CTCC 10b	1.274	0.704	5.7%
	AZ-93 + 6 coats CTCC 10b	1.405	0.835	5.3%
	AZ-93 + 7 coats CTCC 10b	1.550	0.980	5.1%
	AZ-93 + 8 coats CTCC 10b	1.764	1.194	4.8%
	AZ-93 + 9 coats CTCC 10b	1.907	1.337	4.7%
	AZ-93 + 10 coats CTCC 10b	2.112	1.542	4.2%

Table 6. CTCC Formulation 10b on Metal Panels, with and without AZ-93 Paint.

CTCC Formulation 10b on Silver Foil and another Aluminum Panel

A new batch of CTCC Formulation 10b was prepared to test the formulation's performance on silver substrates. A scuffed silver foil, Ag-2, a non-scuffed silver foil, Ag-3, and a prepared aluminum panel, Al-17, were sprayed for comparison.

Figure 57 through Figure 59 show these substrates sprayed with CTCC Formulation 10b after the application of each coat of spray. Al-17 looked similar to other aluminum panels coated with CTCC sprays. Ag-2 and Ag-3 both had some flaking. CTCC Formulation 10b does not adhere to silver as well as it does to aluminum, but similar flaking had been seen in the past with other substrates as well. It is possible that thinner coats could result in some improvement in the coating's adhesion and reduce flaking. The chips in Ag-2's side on the seventh coat were due to measurements made on the sample.



Figure 57. Al-17 with one through ten coats of CTCC Formulation 10b spray.



Figure 58. Ag-2 with one through ten coats of CTCC Formulation 10b spray.



Figure 59. Ag-3 with one through ten coats of CTCC Formulation 10b spray.

Figure 60 through Figure 62 show the spectral results for these samples. Al-17 showed similar reflectance to other aluminum panels coated with CTCC sprays, but with very little change in coats 7-10, suggesting that it may not have continued to improve much with the application of additional coats.

Ag-2 and Ag-3 had very similar reflectance results. It is noteworthy that, at longer wavelengths, the reflectance decreased as the number of coats increases, while at shorter wavelengths, the reflectance increased as the number of coats increases. With aluminum and stainless steel substrates, the reflectance improved across all wavelengths as coats increased. This was most likely due to silver having better reflective properties than aluminum and stainless steel at longer wavelengths.



Figure 60. Reflectance data for Al-17 with CTCC Formulation 10b spray.



Figure 61. Reflectance data for Ag-2 with CTCC Formulation 10b spray.



Figure 62. Reflectance data for Ag-3 with CTCC Formulation 10b spray.

Table 7 lists the resultant solar absorptions for each coat, along with the sample masses following each spray coat. The best result achieved thus far for any combination of substrates and coatings was after ten coats of CTCC Formulation 10b spray on the non-scuffed silver foil sample, at 3.3%.

The scuffed silver foil, Ag-2, had similar performance to aluminum panels previously coated with AZ-93 paint, 4.3%. Its higher solar absorptance than Ag-3 could be due to the scuffing on the surface of the foil.

The aluminum panel, Al-17, had similar results to past aluminum panels coated with AZ-93 paint prior to the addition of CTCC Formulation 10b spray, 4.7% - better than any aluminum panel so far. This was likely due to a larger mass of spray being added to Al-17 than to previous aluminum panels. Al-17 had 2.340 g of spray added, compared to Al-16's 1.611 g of spray which yielded 7.2% solar absorptance.

Sample	Coating	CTCC Mass (g)	Solar Absorption
Al-17	None	0.000	30.4%
	1 coat CTCC 10b	0.147	26.1%
	2 coats CTCC 10b	0.277	19.6%
	3 coats CTCC 10b	0.492	14.6%
	4 coats CTCC 10b	0.726	12.9%
	5 coats CTCC 10b	1.060	7.8%
	6 coats CTCC 10b	1.442	6.1%
	7 coats CTCC 10b	1.866	5.2%
	8 coats CTCC 10b	2.085	4.9%
	9 coats CTCC 10b	2.247	5.0%
	10 coats CTCC 10b	2.340	4.7%
Ag-2	None	0.000	23.6%
	1 coat CTCC 10b	0.017	11.4%
	2 coats CTCC 10b	0.032	8.7%
	3 coats CTCC 10b	0.055	7.2%
	4 coats CTCC 10b	0.082	6.1%
	5 coats CTCC 10b	0.111	5.8%
	6 coats CTCC 10b	0.135	5.7%
	7 coats CTCC 10b	0.181	5.1%
	8 coats CTCC 10b	0.207	4.8%
	9 coats CTCC 10b	0.239	4.7%
	10 coats CTCC 10b	0.302	4.3%
Ag-3	None	0.000	14.5%
	1 coat CTCC 10b	0.019	9.7%
	2 coats CTCC 10b	0.035	7.2%
	3 coats CTCC 10b	0.053	6.1%
	4 coats CTCC 10b	0.077	5.2%
	5 coats CTCC 10b	0.119	4.8%
	6 coats CTCC 10b	0.137	4.5%
	7 coats CTCC 10b	0.165	4.3%
	8 coats CTCC 10b	0.212	4.2%
	9 coats CTCC 10b	0.271	3.3%
	10 coats CTCC 10b	0.306	3.3%

Table 7. CTCC Formulation 10b on aluminum and silver substrates.

CTCC Formulation 10b on Multi-Layer Insulation

CTCC Formulation 10b was also applied to pieces of MLI comprised of ten layers of double-sided aluminum with scrim layers in between. For the first two samples, MLI-1 and MLI-2, 2" x 2" pieces were cut from a larger sheet of MLI. For ease of handling and positioning the samples in the Jasco integrating sphere's sample holder, these were mounted to aluminum panels, taping the corners down with painter's tape. MLI-1 and MLI-2 were sprayed in the horizontal configuration for their first five coats.

That configuration resulted in damage to the applied coating. The edges of the MLI hung over the edges of the aluminum panels, causing some flexing of the MLI during handling and testing. The edges of the MLI were also unsealed, allowing all ten layers to flutter when spray was applied.

To minimize these challenges and improve coating, two more samples were prepared, MLI-3 and MLI-4. For these samples, the pieces of MLI were smaller, so no portion of the MLI would extend beyond the aluminum panel to which it was mounted. The edges of the MLI were also sealed using painter's tape to minimize fluttering.

The last five coats of CTCC Formulation 10b spray on MLI-1 and MLI-2, as well as all ten coats of CTCC Formulation 10b spray on MLI-3 and MLI-4, were applied with the samples mounted vertically and spray applied perpendicular to the sample. This was done with the hope that it would minimize flaking of the coating. In an effort to minimize damage due to handling, these coats were applied without taking reflectance measurements between each coat.

In spite of these efforts, MLI-1 and MLI-2 continued to lose some of their coating in areas that were previously damaged. MLI-3 also had some cracking in one corner which led to a loss of some coating, likely near a defect in the MLI or area where it was uneven after taping the sides of the sample. MLI-4 remained completely coated with no visible damage. Figure 63 through Figure 66 show coats of CTCC Formulation 10b applied to the four MLI samples.



Figure 63. MLI-1 with up to 10 coats of CTCC Formulation 10b - no photo for 7^{th} coat.



Figure 64. MLI-2 with up to 10 coats of CTCC Formulation 10b - no photo for 7^{th} coat.



Figure 65. MLI-3 with 1-10 coats of CTCC Formulation 10b spray – no photo for 2^{nd} coat.



Figure 66. MLI-4 with 1-10 coats of CTCC Formulation $10b - no photo for 2^{nd} coat$.

Figure 67 through Figure 70 show the reflectance results for the MLI samples. Further investigation is still required to determine why MLI-1 and MLI-2 both showed a drop in reflectance after the first coat of CTCC Formulation 10b spray was added; however, both samples showed improvement in reflectance over the bare MLI after a second coat was added, creating more even coverage. By the tenth coat, all four MLI samples coated with CTCC spray showed significant improvement in reflectance.



Figure 67. Reflectance data for MLI-1 coated with CTCC Formulation 10b spray.



Figure 68. Reflectance data for MLI-2 coated with CTCC Formulation 10b spray.



Figure 69. Reflectance data for MLI-3 coated with CTCC Formulation 10b spray.



Figure 70. Reflectance data for MLI-4 coated with CTCC Formulation 10b spray.

Table 8 shows the solar absorptions for all coats for which it was measured. Sample masses are also included, but should not be considered an accurate reflection of the accumulated mass of coating on the MLI due to the added mass of overspray onto the tape and aluminum panels as well as coating loss due to damage.

All of the MLI samples had solar absorptance values in the 4-5% range. Slightly thinner coats of spray were used in coats 6-10 of MLI-1 and MLI-2 as well as all ten coats on MLI-3 and MLI-4 in the hope of reducing flaking. The likely thicker coating on MLI-1 and MLI-2 is the presumed cause of those two samples having lower solar absorptances.

Sample	Coating	CTCC Mass (g)	Solar Absorption
MLI-1	None	0.000	9.55%
	1 coat CTCC 10b	0.070	13.21%
	2 coats CTCC 10b	0.148	9.00%
	3 coats CTCC 10b	0.291	6.05%
	4 coats CTCC 10b	0.475	4.97%
	5 coats CTCC 10b	0.712	4.70%
	10 coats CTCC 10b	0.837	4.35%
MLI-2	None	0.000	9.84%
	1 coat CTCC 10b	0.060	12.88%
	2 coats CTCC 10b	0.131	9.26%
	3 coats CTCC 10b	0.255	7.80%
	4 coats CTCC 10b	0.397	6.67%
	5 coats CTCC 10b	0.635	4.59%
	10 coats CTCC 10b	0.662	4.14%
MLI-3	None	0.000	10.11%
	10 coats CTCC 10b	1.273	4.71%
MLI-4	None	0.000	10.33%
	10 coats CTCC 10b	1.074	4.89%

Table 8. CTCC Formulation 10b on MLI substrates.

CTCC Formulation 10b on Aluminum Discs

In addition to the aluminum Q-panels, discs of 7075 aluminum with a machined finish were sprayed with CTCC Formulation 10b. It was expected that a smoother finish would have better solar absorption, but may not result in the best adhesion of the spray to the surface. To investigate both options, one disc, C-1, was degreased but not scuffed, and another, C-2, was degreased, scuffed, and degreased again – following the same process used for the aluminum and stainless steel coupons.

Both discs had good spray adhesion. A rudimentary adhesion check was also performed on the overspray which coated the (unscuffed) outer edge of one disc by rubbing it with a gloved finger. While a small amount of the coating did transfer to the glove, the majority of the coating remained intact.

Figure 71 and Figure 72 show C-1 and C-2 sprayed with CTCC Formulation 10b.



Figure 71. Aluminum disc C-1 before coating and after each coat of CTCC Formulation 10b.



Figure 72. Aluminum disc C-2 before coating and after each coat of CTCC Formulation 10b spray.

Figure 73 and Figure 74 show reflectance data collected for C-1 and C-2 before spraying and after the addition of each coat of CTCC Formulation 10b spray. Both samples showed improvement as coats of spray were added. There was a sizeable improvement after just one coat of spray. Coats 7-10 had similar reflectivities in the visible wavelengths, but those coats did continue to improve in the ultraviolet.



Figure 73. Reflectance data for aluminum disc C-1 coated with CTCC Formulation 10b spray.



Figure 74. Reflectance data for aluminum disc C-2 coated with CTCC Formulation 10b spray.

Table 9 gives the solar absorptance and coating mass of C-1 and C-2 prior to spraying and after the addition of each coat of spray. The coating masses do include the overspray that accumulated on the sides of the samples, so they are not representative of the mass of the coated surface alone.

These samples have the lowest solar absorptances of any CTCC-coated samples thus far, with C-1 at only 2.8%. C-2 has a slightly higher solar absorptance than C-1 in spite of having more mass of spray added overall. This could be because C-2 accumulated more overspray and has a lower coating mass on the analyzed top surface, but it is also possible that the smoother, unscuffed surface of C-1 led to its lower solar absorption.

Sample	Coating	CTCC Mass (g)	Solar Absorption
C-1	None	0.000	29.8%
	1 coat CTCC 10b	0.013	16.9%
	2 coats CTCC 10b	0.032	11.5%
	3 coats CTCC 10b	0.061	9.0%
	4 coats CTCC 10b	0.096	7.6%
	5 coats CTCC 10b	0.147	6.1%
	6 coats CTCC 10b	0.171	5.8%
	7 coats CTCC 10b	0.268	4.3%
	8 coats CTCC 10b	0.303	3.5%
	9 coats CTCC 10b	0.339	3.0%
	10 coats CTCC 10b	0.357	2.8%
C-2	None	0.000	33.5%
	1 coat CTCC 10b	0.016	21.4%
	2 coats CTCC 10b	0.033	15.8%
	3 coats CTCC 10b	0.067	12.5%
	4 coats CTCC 10b	0.095	11.0%
	5 coats CTCC 10b	0.151	8.1%
	6 coats CTCC 10b	0.175	7.4%
	7 coats CTCC 10b	0.272	5.1%
	8 coats CTCC 10b	0.318	4.2%
	9 coats CTCC 10b	0.350	3.8%
	10 coats CTCC 10b	0.369	3.6%

Table 9. CTCC Formulation 10b on machined 7075 aluminum.

Spray Coating on Rigid Y₂O₃

While spray coating on metal samples, CCTCC Formulation 9 was also applied to some of the Y_2O_3 rigid samples. This allowed for the evaluation of the spray coating without the impact of the metal backing. Similarly, some Y_2O_3 rigid samples were also painted with AZ-93 and then spray coated with CTCC Formulation 9 to determine the overall impact of the metal backing on spectral data for those samples as well.

Sample Preparation

Details of the fabrication of rigid Y_2O_3 samples is included in Appendix C.

Rigid Y_2O_3 samples 20Y-13, 20Y-16, 20Y-22, 20Y-27, 20Y-28, 20Y-29, 20Y-30, 20Y-31, 20Y-32, and 20Y-34, were painted with AZ-93 following the same methods for mixing, spraying, and curing used for the metal samples with AZ-93 paint basecoats.

Spectral analysis was performed on these samples following the same methods used for spray coat analysis to determine what, if any, impact was created by painting a rigid Y_2O_3 sample with AZ-93 paint, rather than the traditional metal substrate.

Rigid Y₂O₃ samples 20Y-35, 20Y-36, and 20Y-37, and AZ-93 painted rigid Y₂O₃ samples 20Y-13, 20Y-16, 20Y-27, 20Y-28, and 20Y-29, were then spray coated with CTCC Formulation 9, following the same methods used for spraying CTCC Formulation 9 onto metal substrates. Figure 75 through Figure 82 show these

samples with their coatings. Some of the CTCC Formulation 9 spray, or possibly AZ-93 paint and CTCC Formulation 9 spray, delaminated from the surface due to being optimized for coating metal, not a chalky material like the Y_2O_3 substrate.



Figure 75. Sample 20Y-13 coated with AZ-93 paint, then by 1 and 2 coats of CTCC Formulation 9 spray.



Figure 76. Sample 20Y-16 coated with AZ-93 paint, then by 1 and 2 coats of CTCC Formulation 9 spray.



Figure 77. Sample 20Y-27 coated with AZ-93 paint, then 1 and 2 coats of CTCC Formulation 9 spray.



Figure 78. Sample 20Y-28 coated with AZ-93 paint, then 1 and 2 coats of CTCC Formulation 9 spray.



Figure 79. Sample 20Y-29 coated with AZ-93 paint, then 1 and 2 coats of CTCC Formulation 9 spray.



Figure 80. Sample 20Y-35 sprayed with 1 and 2 coats of CTCC Formulation 9.



Figure 81. Sample 20Y-36 sprayed with 1 and 2 coats of CTCC Formulation 9.



Figure 82. Sample 20Y-37 sprayed with 1 and 2 coats of CTCC Formulation 9.

Figure 83 shows an example of reflectance data for CTCC Formulation 9 spray over a rigid Y_2O_3 sample. While the first coat did show some decrease in reflectivity, possibly due to the KBr binder used in the spray, the results were very similar to those of the Y_2O_3 rigid sample alone after the second coat of spray. The bulk of the difference between the two was in the region of the spectrum which has been determined to be related to absorption from water on the surface or within the sample. This demonstrates that the spray coating does not significantly differ from the rigid Y_2O_3 materials.



Figure 83. Reflectance data for CTCC Formulation 9 spray over a Y₂O₃ rigid sample, 20Y-35.

Figure 84 shows an example of reflectance data for one of the rigid Y₂O₃ samples which was coated with AZ-93 paint, and then sprayed with CTCC Formulation 9. Results were similar for similarly coated samples.

Adding AZ-93 paint to the rigid Y_2O_3 samples resulted in a decrease of reflectance, particularly in the ultraviolet range. The AZ-93 paint's performance on the Y_2O_3 samples was very similar to the results for AZ-93 paint on metals.

Spraying the AZ-93 painted Y_2O_3 samples brought the reflectance back up to nearly that of the Y_2O_3 samples prior to being painted.



Figure 84. Reflectance data for AZ-93 paint and CTCC Formulation 9 spray on a rigid Y₂O₃ *sample.*

Table 10 lists calculated solar absorption values for each coat.

AZ-93 paint had better solar absorptions on Y_2O_3 than it did on metals. While the AZ-93 paint thickness met the manufacturer's recommendations when applied, it is possible that the AZ-93 paint was not thick enough to eliminate the impacts of the metal substrates, which absorb more light than the Y_2O_3 substrate does.

Without the absorption of the metal substrates, CTCC Formulation 9 spray achieves better results as well. When sprayed over AZ-93 paint on a Y_2O_3 substrate, the solar absorption for CTCC Formulation 9 reaches 2-3% in only two coats. However, this drop of roughly 6% from the measured absorption of the AZ-93 paint basecoat was similar to the improvement in solar absorption measured after two coats of CTCC Formulation 9 spray were added to metal substrates with AZ-93 basecoats.

This suggests that the metal substrates were still impeding the results seen for the CTCC sprays on aluminum and stainless steel and that, with thicker overall coatings (more coats), even lower solar absorption values could be reached.

Sample	Coating	Solar Absorption
20Y-35	None	2.28%
	1 coat CTCC 9	4.02%
	2 coats CTCC 9	3.31%
20Y-36	None	2.19%
	1 coat CTCC 9	6.76%
	2 coats CTCC 9	4.10%
20Y-37	None	1.92%
	1 coat CTCC 9	4.37%
	2 coats CTCC 9	3.33%
20Y-22	None	0.71%
	AZ-93	8.31%
20Y-30	None	0.97%
	AZ-93	8.67%
20Y-31	None	3.46%
	AZ-93	11.81%
20Y-32	None	4.59%
	AZ-93	10.57%
20Y-34	None	3.32%
	AZ-93	10.77%
20Y-13	None	1.13%
	AZ-93	8.81%
	AZ-93 + 1 coat CTCC 9	3.43%
	AZ-93 + 2 coats CTCC 9	2.13%
20Y-16	None	10.2%
	AZ-93	9.37%
	AZ-93 + 1 coat CTCC 9	4.08%
	AZ-93 + 2 coats CTCC 9	2.21%
20Y-27	None	1.38%
	AZ-93	9.58%
	AZ-93 + 1 coat CTCC 9	4.22%
	AZ-93 + 2 coats CTCC 9	10.54%
	(CTCC spray delaminated)	
20Y-28	None	0.79%
	AZ-93	9.14%
	AZ-93 + 1 coat CTCC 9	4.39%
	AZ-93 + 2 coats CTCC 9	2.57%
20Y-29	None	0.84%
	AZ-93	8.24%
-	AZ-93 + 1 coat CTCC 9	3.41%
	AZ-93 + 2 coats CTCC 9	2.29%

Table 10. Solar Absorption for Rigid Y₂O₃ Samples with AZ-93 Paint and/or CTCC Formulation 9 Spray.

Impact of Water Absorption on Sample Data

The dips in reflectance that appear in all spectral data at wavelengths greater than 1300 nm have been determined to be water absorption peaks. Appendix D gives details of testing performed to verify this.

In the vacuum of space, the water should outgas, eliminating the water absorption peaks seen in the lab and improving the solar absorption of the coatings. To better estimate the solar absorption of the coatings without water, solar absorption values were recalculated after removing water absorption peaks from the data. These calculations are very conservative, only replacing the main water absorption peaks in the original data with linear functions extending from one end of the peak's location to the other. Figure 85 through Figure 92 show reflectance data for several substrates coated with CTCC Formulation 10b spray that have had their water absorption peaks removed in this manner.

Completely drying the samples would likely result in some additional increases in reflectance and decreases in their solar absorptions.



Figure 85. Reflectance data for Al-11 with a flat white spray paint basecoat and CTCC Formulation 10b spray, with water absorption peaks removed. Original data are shown in Figure 43.



Figure 86. Reflectance data for Al-16 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 54.



Figure 87. Reflectance data for SS-14 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 55.



Figure 88. Reflectance data for Al-4 with an AZ-93 paint basecoat and CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 56.



Figure 89. Reflectance data for Ag-3 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 62.



Figure 90. Reflectance data for Al-17 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 60.



Figure 91. Reflectance data for MLI-2 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 68.


Figure 92. Reflectance data for C-1 sprayed with CTCC Formulation 10b, with water absorption peaks removed. Original data are shown in Figure 73.

Table 11 gives solar absorption values before and after water peak removal for select samples. The improvement in solar absorption with water peak removal ranges from 0.1% to 0.3%, with a typical value of 0.2%. While this does not represent a large change, it could result in significant improvement in thermal properties of spacecraft or other objects coated with CTCC spray.

Sample	Coating	Solar Absorption	Solar Absorption –
			Water Peaks Removed
Al-11	Flat white spray paint	29.0%	28.9%
	Paint + 1 coat CTCC 10b	19.2%	19.0%
	Paint + 2 coats CTCC 10b	11.0%	10.9%
	Paint + 3 coats CTCC 10b	9.4%	9.3%
	Paint + 4 coats CTCC 10b	10.1%	10.0%
Al-16	1 coat CTCC 10b	27.6%	27.5%
	2 coats CTCC 10b	23.9%	23.8%
	3 coats CTCC 10b	18.2%	18.0%
	4 coats CTCC 10b	13.8%	13.6%
	5 coats CTCC 10b	12.0%	11.8%
	6 coats CTCC 10b	10.5%	10.3%
	7 coats CTCC 10b	10.4%	10.2%
	8 coats CTCC 10b	7.8%	7.6%
	9 coats CTCC 10b	7.6%	7.3%

Table 11. Solar Absorption of Selected Samples with and without Water Peaks Removed.

	10 coats CTCC 10b	7.2%	7.0%
SS-14	1 coat CTCC 10b	37.1%	37.0%
	2 coats CTCC 10b	28.6%	28.5%
	3 coats CTCC 10b	24.3%	24.1%
	4 coats CTCC 10b	16.5%	16.3%
	5 coats CTCC 10b	12.3%	12.1%
	6 coats CTCC 10b	10.6%	10.4%
	7 coats CTCC 10b	9.7%	9.5%
	8 coats CTCC 10b	9.5%	9.3%
	9 coats CTCC 10b	8.8%	8.6%
	10 coats CTCC 10b	7.2%	7.0%
Al-4	AZ-93 Paint	13.9%	13.8%
	AZ-93 + 1 coat CTCC 10b	10.3%	10.1%
	AZ-93 + 2 coats CTCC 10b	8.0%	7.8%
	AZ-93 + 3 coats CTCC 10b	7.2%	6.9%
	AZ-93 + 4 coats CTCC 10b	6.6%	6.3%
	AZ-93 + 5 coats CTCC 10b	5.7%	5.5%
	AZ-93 + 6 coats CTCC 10b	5.3%	5.1%
	AZ-93 + 7 coats CTCC 10b	5.1%	4.8%
	AZ-93 + 8 coats CTCC 10b	4.8%	4.6%
	AZ-93 + 9 coats CTCC 10b	4.7%	4.5%
	AZ-93 + 10 coats CTCC 10b	4.2%	3.9%
Ag-3	1 coat CTCC 10b	9.7%	9.5%
	2 coats CTCC 10b	7.2%	7.0%
	3 coats CTCC 10b	6.1%	5.9%
	4 coats CTCC 10b	5.2%	5.0%
	5 coats CTCC 10b	4.8%	4.6%
	6 coats CTCC 10b	4.5%	4.3%
	7 coats CTCC 10b	4.3%	4.1%
	8 coats CTCC 10b	4.2%	4.0%
	9 coats CTCC 10b	3.3%	3.0%
	10 coats CTCC 10b	3.3%	3.0%
Al-17	1 coat CTCC 10b	26.1%	26.0%
	2 coats CTCC 10b	19.6%	19.4%
	3 coats CTCC 10b	14.6%	14.5%
	4 coats CTCC 10b	12.9%	12.7%
	5 coats CTCC 10b	7.8%	7.6%
	6 coats CTCC 10b	6.1%	5.9%
	7 coats CTCC 10b	5.2%	5.0%
	8 coats CTCC 10b	4.9%	4.7%
	9 coats CTCC 10b	5.0%	4.8%
	10 coats CTCC 10b	4.7%	4.5%
MLI-2	1 coat CTCC 10b	12.9%	12.8%
	2 coats CTCC 10b	9.3%	9.1%
	3 coats CTCC 10b	7.8%	7.6%
	4 coats CTCC 10b	6.7%	6.5%

	5 coats CTCC 10b	4.6%	4.3%
	10 coats CTCC 10b	4.1%	3.9%
C-1	1 coat CTCC 10b	16.9%	16.8%
	2 coats CTCC 10b	11.5%	11.4%
	3 coats CTCC 10b	9.0%	8.8%
	4 coats CTCC 10b	7.6%	7.4%
	5 coats CTCC 10b	6.1%	5.9%
	6 coats CTCC 10b	5.8%	5.5%
	7 coats CTCC 10b	4.3%	4.0%
	8 coats CTCC 10b	3.5%	3.2%
	9 coats CTCC 10b	3.0%	2.8%
	10 coats CTCC 10b	2.8%	2.5%

Conclusions

The most recent spray-on Y_2O_3 coating, CTCC Formulation 10b, testing shows that this coating can perform better than the current commercial options, in terms of reflectivity and solar absorption. As the only coating, CTCC Formulation 10b has been measured to have solar absorptances as low as 2.8% on aluminum, 7.2% on stainless steel, and 3.3% on silver.

In addition, it can significantly improve performance when sprayed over materials coated with commercially available reflective paint, such as AZ-93, even with only a single coat. The best results at this point for spraying CTCC Formulation 10b over an AZ-93 paint basecoat improved solar absorption from 14% for AZ-93 alone to 10% with a single coat of CTCC spray and to only 4% after ten coats of CTCC spray.

Beyond its improved solar absorption, CTCC Formulation 10b presents several other improvements over the commercial options in both ease of use and cost. CTCC Formulation 10b requires little preparation after mixing and can air dry within hours without requiring any special equipment or chambers. While its shelf life is not yet known, the same bottle of CTCC liquid has been used for multiple weeks after mixing, with only a brief shaking before application. In addition, this coating is expected to be a cheaper option than others on the market.

Overall, CTCC Formulation 10b spray is a very promising option for future space applications.

Future Work

As mentioned throughout this report, additional efforts in optimization will lead to improved performance. These efforts may include the following areas:

- Continue optimizing CTCC formulations.
- Continue optimizing spray methods.
- Continue optimizing amount of spray to apply.
- Re-test previous materials with more optimized spraying to determine peak performance across all materials.
- Determine minimum drying time between sprays to optimize production.
- Perform additional testing for water removal to determine potential performance improvement in space more accurately.
- Test spraying on larger surfaces.

• Investigate improvement with other basecoat paints.

Appendix A - Issues with Measuring Solar Absorption Using Reflectometry

The standard approach for measuring solar absorptivity is described in ASTM E903, "Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres" and is also described in Ref. [1]. From these documents, solar absorption is calculated by measuring the reflectance of a material using a spectrometer with an integrating sphere over some spectral range (typically 300 nm to 2400 nm, though some companies, such as Xioptiq integrate from 250 nm to 2500 nm).

Let the measured reflectivity versus wavelength be given by $\rho(\lambda)$, a value between 0 and 1. Then the spectral absorptivity is given by $\alpha(\lambda) = 1 - \rho(\lambda)$ and, from [1], the total solar reflectivity, ρ , or the total solar absorptivity, α , is given by



where $\alpha = 1 - \rho$ and where $I_s(\lambda)$ is the solar spectral irradiance in deep space at 1 A.U. from the Sun. The solar spectral irradiance, $I_s(\lambda)$, is given by ASTM E-490 and is plotted in the figure below.



Figure 93. The deep space spectral irradiance of the Sun.

This method for measuring solar absorptivity is reasonable if the reflectors have moderate absorptance and small offsets in the measurement are permitted. However, if solar reflectors with very low absorptance are desired then this approach may yield misleading results due to the limited range of measurement, the relative nature of the measurement, and water contamination.

Limited Range of Measurement

The ASTM E903 approach for measuring solar absorptivity only considers sunlight over the 300 nm to 2400 nm range. However, there is substantial solar power outside of this range as shown in the plots below. About 1.1 % of the Sun's total irradiance is below 300 nm (about 0.155 % below 250 nm). About 3.7% of the Sun's total irradiance is beyond 2400 nm and 3.3% beyond 2500nm.



Figure 94. The total percentage of Solar Irradiance below a given wavelength in the ultraviolet region of the spectrum.



Figure 95. The total percentage of Solar Irradiance below a given wavelength in the infrared region of the spectrum.

So the integral band used in Ref [4] leaves out about 4.8% of the Sun's power, most of which is in bands where many materials, such as plastics, organic binders, and standard optical scatterers, absorb radiation. As an example, consider the reflectance spectrum of a common spacecraft white coating, AZ-93, shown in the figure. This material is advertised as having a solar absorptivity of about 14%, but this is over a limited range. This material absorbs short wave radiation below 300 nm and likely has IR absorbance, so its actual solar absorptivity is likely closer to 17-18%.



Figure 96. Spectral Reflectance of AZ-93 on aluminum.

In our work, we integrate from 200 nm to 2400 nm to calculate the solar absorptivity because the spectrometers being used extend over that region.

Comparison with Spectralon

Another problem with using spectral reflectivity measurements is that the spectrometer has to be calibrated to a reflectance standard, which itself is not a perfect reflector. The material typically used for this is Spectralon and its reflectivity (from the manufacturer) is shown in the plot below compared against yet another reference standard, pressed PTFE.



Figure 97. Spectral Reflectivity of NIST Spectralon sample.

The total offset introduced by the reference standard is about 1%, based on the NIST data supplied with some certified reflectors we recently purchased.

Water

Another problem with measuring solar absorption is that the performance of the reflector is affected by the presence of water. Water has strong absorption bands at about 1400 nm and 1900 nm. The figure below shows this absorption for a 12 micron thick layer of water, which is a significant amount of water. However, the reflectors under consideration are composed of scatterers where light can interact thousands of times with the scattering particles before making its way back out of the coating. Consequently, even a layer of water three molecules thick coating the particles, can result in an interaction with a layer of water that is effectively a few microns thick.

So a measurement of solar absorptivity in a humid environment can be offset negatively compared with the dry environment of deep space, assuming the water can escape, i.e. that it is not highly bound or trapped by the coating. Both the AZ-93 and Spectralon spectra shown above have absorption dips in the infrared that are likely due to water.



Figure 98. The transmission of light through 12 microns of water.

Appendix B – Correction for Varying Baselines

At the beginning of this project, data were collected using a Jasco V-670 and its ISN-723 integrating sphere accessory. Baseline corrections for these data used a Spectralon[®] sample which had been provided by Jasco when the instrument was purchased many years ago. When a new instrument, the Jasco V-770, was purchased, a new Spectralon[®] sample was provided. Data collected using the new Spectralon[®] for the baseline should have been identical to data collected using the old Spectralon[®] for the baseline, but the results differed greatly.

After several comparisons were made, a NIST-traceable Spectralon[®] was purchased and compared to the others. While some differences were notable between varying grades of Spectralon[®], it became clear that the older Spectralon[®] had either become contaminated or degraded during its many years of use.

Figure 99 shows data collected for both the NIST-traceable Spectralon[®] (SN 3153) and the old Spectralon[®], using the NIST-traceable Spectralon[®] as the baseline.



Figure 99. Reflectance data showing results for an old Spectralon[®] sample and a new, NIST-traceable Spectralon[®] sample, indicating degradation or contamination in the old sample.

The NIST-traceable Spectralon[®] sample was used as the baseline for all spectral measurements collected afterward, but some of the data for the coating study could not easily be reproduced. As such, a conversion to make older data comparable to the newer data was necessary. Theory indicated that the following calculation, applied to each data point, should yield equivalent data:



This theory was tested using several datasets, comparing the resultant data from the above calculation for sample data collected using the old Spectralon[®] to data collected for the same sample using the new, NIST-traceable Spectralon[®]. The results indicated that this method, indeed, would yield equivalent data.

Figure 100 and Figure 101 show a comparison between data collected with the NIST-traceable Spectralon[®] as the baseline and the equivalent data calculated from data collected for the same sample with old Spectralon[®] as the baseline, showing a good match.



Figure 100. Reflectance data comparing original data for a Y₂O₃ sample collected using a new Spectralon[®] baseline to equivalent data calculated from sample data collected with an old Spectralon[®] baseline.



Figure 101. The same data shown in Figure 100, zoomed in on a smaller region.

After verifying this method, equivalent datasets were calculated for all coating study data that had been collected using the old Spectralon[®].

Appendix C – Rigid Y₂O₃ Sample Fabrication

The methods for fabrication of rigid Y_2O_3 samples have evolved over time. Changes include mold type, pressure applied, amount of water added, and fabrication tools used. The rigid Y_2O_3 samples used for the coating study were made over several weeks, with slight variations in method.

The current fabrication procedure and best practices are listed below. Table 12 includes specific details about the fabrication methods for each sample used in the coating study.

Current Fabrication Procedure

- 1. All steps of this procedure should be performed wearing clean gloves and taking care to avoid contamination.
- 2. Clean the workspace.
 - Remove any nearby clutter, particularly any materials that could shed particles and contaminate samples.
 - Wipe preparation surfaces with a damp cloth to remove dust, or clean more thoroughly as needed. Allow to fully dry before continuing.
 - Place a clean non-linting wipe on the work surface.
- 3. Clean all equipment.
 - To reduce contamination, dedicated equipment for use with Y₂O₃ sample fabrication is recommended.
 - Clean all equipment used for fabrication with tap water. Use a scrubber as needed.
 - Rinse all equipment used for fabrication with high purity water.
 - Dry non-porous equipment with a non-linting cloth. Cover until ready for use.
 - Allow ceramic molds to dry overnight, covered with a non-linting cloth.
- 4. Clean the workspace again.
- 5. Tare a watch glass or other, small, glass container on a balance.
 - Plastic or metal weigh dishes can be used, but risk contaminating the Y_2O_3 .
- 6. Add desired amount of high purity Y_2O_3 powder to the watch glass using a metal spatula.
 - In the current process, ~2.9 g to ~3.5 g of Y_2O_3 is used to fabricate samples that are ~2.9 mm to ~3.8 mm thick with 65-67% void volume.
- 7. Tare the balance.
- 8. Using a clean dropper, add high purity water to the Y_2O_3 on the balance. Total mass of high purity water added should be ~10% of the Y_2O_3 mass.
- 9. Remove the watch glass from the balance and mix the Y_2O_3 and water with a metal spatula.
 - Use a chopping motion and scatter the material to aid in distributing the water throughout the mixture since it typically sticks to the yttrium oxide upon addition
 - Move the material into a pile again and repeat chopping and scattering several times, until no large clumps form and the material is thoroughly mixed.
- 10. Using a metal spatula, carefully add all of the Y_2O_3 -water mixture to one of the molds.
 - Avoid scraping Y_2O_3 that lands on the top of the mold inside, as this may cause contamination.
 - Add some of the Y_2O_3 -water mixture, then gently press it down with the spatula to make room for more.
 - When gently pressing with a spatula does not create enough room, place the sapphire disk into the mold and gently press down (by hand) to compress the Y₂O₃-water mixture

into the mold, creating more space. Be sure that the sapphire disk does not touch the sides of the mold's inner surface, as this could break away pieces of ceramic and contaminate the sample.

- 11. When the Y₂O₃-water mixture has been added to the mold, place the sapphire disk over it, being careful to center the disk on the mold's opening so that it does not touch the sides.
- 12. Gently push down on the sapphire disk so that it is just below the top edge of the mold.
 - This prevents the sapphire disk from moving when the mold is placed in the press.
 - Check to ensure that the top of the sapphire disk is level and adjust as needed. This will reduce the disk scraping the sides of the mold during pressing and contaminating the sample.
- 13. Gently blow or brush away the Y₂O₃-water mixture that has fallen onto the top surface of the mold, now that the sapphire disk will prevent it from falling into the mold's opening.
- 14. Sit the mold (with sapphire disk in place) in the press.
- 15. Drape a small, non-linting cloth over the sapphire disk and mold to prevent anything from falling onto the surface during pressing.
- 16. Using the press, apply force, compressing the sapphire disk into the mold.
 - Currently, a force of 250 kg is applied.
- 17. Hold under pressure for 2 minutes.
- 18. Remove the mold from the press.
- 19. Carefully remove the sapphire disk.
 - Sit the mold down on the benchtop.
 - Press down on the top of the mold to hold it in place.
 - Gently rotate the sapphire disk, keeping it level, to break it free from the $Y_2O_3\mbox{-water}$ mixture in the mold.
 - Pull the sapphire disk up and out of the mold.
- 20. Gently blow on the Y_2O_3 -water mixture in the mold to remove some of the loose material that has fallen onto its surface after removing the sapphire disk.
- 21. Allow the Y_2O_3 -water mixture to rest in the mold overnight, covered with a non-linting cloth, before sintering.
- 22. Sinter the Y_2O_3 -water mixture in the mold.
 - The sintering process is undergoing optimization, but sintering profile used for the coating study samples is described below.
- 23. Gently pull the top of the mold off of the bottom and remove the sample from the mold.
 - The sample is most likely going to be stuck in the top piece of the mold. If so, gently tap it with a gloved finger to remove it.
- 24. Remove any raised edges from the sample by gently sanding with a fine, stainless steel wire mesh, being especially careful not to touch the center of the sample's surface.

Current Sintering Process

- 1. Place the samples, still in their molds, in the furnace.
- 2. Ramp up from room temperature to 250°C.
- 3. Hold at 250°C for 45 minutes.
- 4. Ramp up from 250°C to 500°C.
- 5. Hold at 500°C for 45 minutes.
- 6. Ramp up from 500°C to 750°C.

- 7. Hold at 750°C for 45 minutes.
- 8. Ramp up from 750°C to 1000°C.
- 9. Hold at 1000°C for 45 minutes.
- 10. Ramp up from 1000°C to 1200°C.
- 11. Hold at 1200°C for 90 minutes.
- 12. Turn off furnace, but leave the door closed.
- 13. Allow samples to cool undisturbed in the furnace as it gradually returns to room temperature.
- 14. Samples should cool for several hours, until they are cool enough to handle.

Sample	Mold	Pressing Force (kg)	Thickness (mm)	Void Volume
20Y-13	1" Aluminum	500	2.3	61.9%
20Y-16	1" Aluminum	500	2.4	63.9%
20Y-22	1" Aluminum	500	2.1	59.7%
20Y-27	0.8" Ceramic	250	3.9	63.4%
20Y-28	0.8" Ceramic	250	3.8	62.3%
20Y-29	0.8" Ceramic	250	4.0	64.1%
20Y-30	0.8" Ceramic	250	3.8	63.5%
20Y-31	0.8" Ceramic	250	4.4	67.3%
20Y-32	0.8" Ceramic	250	4.0	64.4%
20Y-34	0.8" Ceramic	250	4.0	64.5%
20Y-35	0.8" Ceramic	250	3.9	63.6%
20Y-36	0.8" Ceramic	250	3.9	63.6%
20Y-37	0.8" Ceramic	250	3.7	61.6%

Table 12. Details on fabrication of rigid Y_2O_3 samples used in the coating study.

Appendix D – Water Removal Testing

Spectral analysis of both rigid Y_2O_3 samples and samples sprayed with Y_2O_3 -based sprays, such as CTCC Formulations 9, 10, and 10b, all show dips in reflectance at wavelengths above 1300 nm. Based on the known absorption of water, these dips were thought to be water absorption peaks.

In order to test this theory, spectral measurements were made after adding water to and removing water from samples to observe shifts in their reflectance. The measurements for adding water to the sample were made using the Jasco V-770 and its ISN-923 integrating sphere accessory. The measurements for drying samples were made on the Jasco V-670 using its ISN-723 integrating sphere accessory. Both instruments used the following parameters:

- Range = 200-2400 nm
- Data interval = 1 nm
- UV/Vis bandwidth = 2.0 nm
- NIR bandwidth = 20.0 nm
- Response time = 0.24 s
- Scan speed = 1000 nm/min
- Dark and baseline corrections

In order to dry samples, a tube with a flared fitting on its end was positioned just above the sample window of the integrating sphere. The sample being dried was positioned slightly back from the window of the integrating sphere, allowing a gap where dry nitrogen gas (99.999%) flowing through the tube would blow across the surface of the sample. Figure 102 shows diagrams of the test setup. Gas flow was adjusted so that it was just strong enough to be felt by hand at the bottom of the sample chamber.



Figure 102. Front and side views of the test setup for drying samples.

In a perfect setup, the position of the front face of the Spectralon[®] and the position of the front face of the samples, relative to the integrating sphere, should be identical. Effort was made to reproduce the same gap size each time, but slight variations may have occurred in the test setup for drying samples. Because of this, the overall reflectance values, relative to the 100% value set by the Spectralon[®] baselines, may be slightly higher or lower. The focus in this testing was on the relative shift of the water absorption peaks compared to the rest of the reflectance values for a particular dataset rather than on specific reflectance values.

Test 1 – Adding Water

In order to quickly determine if the peaks seen in reflectance data were related to water, two water drops (0.078 g total) were added to a rigid Y_2O_3 sample, 20Y-49, and data were compared. Figure 103 shows the reflectance data for this sample before and after the addition of the water drops, as well as over the next two days.

The change to the reflectance data after adding the water was dramatic. Reflectance was lowered from around 900 nm to 2500 nm, with significant changes in a series of peaks. The dominant peaks were centered at 1450 nm and 1930 nm, areas in which dips in reflectance data seen in samples had previously been theorized to be due to water absorption. This clearly confirmed that theory.

Approximately 20 hours after adding the water, the sample reflectance for 20Y-49 was collected again. In that timeframe, the sample sat under ambient conditions in a covered dish. At that point, the reflectance data were about half way back to their pre-wetting values.

The following day, reflectance data were collected for 20Y-49 one more time. This time, in addition to sitting for another day under ambient conditions, 20Y-49 had also been dried by blowing nitrogen gas across its face for just over an hour. This time, the reflectance curve was a much closer match to the sample's pre-wetting values, but had not entirely returned to its original values.



Figure 103. Reflectance data for Y_2O_3 sample 20Y-49 before and after water drops were added.

Test 2 – Drying Spectralon®

As an initial test of the sample drying process, a piece of NIST-traceable Spectralon[®] (SN 3153) was used for the instrument's baseline measurement. The same Spectralon[®] was then run as a sample, while drying, to see if inverted water peaks appeared as the sample shifted away from the baseline.

Procedure

- 1. Collect the instrument baseline using Spectralon[®].
- 2. Collect reflectance data for Spectralon[®] prior to gas flow.
- 3. Begin gas flow to dry the Spectralon[®].
- 4. Collect reflectance data for the Spectralon[®] after 5 and 10 minutes of gas flow.

Results

Figure 104 shows the reflectance data for the Spectralon[®] sample for each time point. Small, inverse water peaks were apparent in the ranges of 1300-1400 nm and 1800-1900 nm, as well as an overall upward shift in the reflectance across all wavelengths. The peaks confirm that water was present on the Spectralon[®] and that drying will reduce the absorption peaks seen in sample reflectance data.



Figure 104. Reflectance data for a Spectralon[®] sample dried with nitrogen gas blowing across its surface.

Test 3 – Drying a Rigid Y₂O₃ Sample

After confirming that the drying process was effective with the Spectralon[®] test, a new test was conducted to observe water removal from a rigid Y_2O_3 sample, 20Y-49. This was the same sample which had water droplets added to it one day beforehand.

Procedure

- 1. Begin nitrogen gas flow to dry the Spectralon[®].
- 2. Collect a baseline using NIST-traceable Spectralon[®] dried with nitrogen gas for 15 minutes.
- 3. Turn off nitrogen gas flow.
- 4. Collect reflectance data for 20Y-49 prior to gas flow.
- 5. Begin nitrogen gas flow to dry the sample.
- 6. Collect reflectance data for 20Y-49 at t = 5, 10, 15, 20, 25, 30, 60, and 65 minutes of gas flow.
- 7. Turn off gas flow.

Results

Figure 105 shows the reflectance data for 20Y-49 for each time point. The reflectance rises across all wavelengths with each measurement, but the increases become smaller over time. This could be partially due to the sample moving forward slightly while drying, as this sample was not as well affixed as the Spectralon[®] or Y₂O₃ samples in later test runs. Some upward shift was also seen in the reflectance data of Spectralon[®] in the previous and following tests, however, when the Spectralon was secured in place and not moving, so other factors, such as refraction due to water on the surface of the sample, also played a part. Further testing would be required to fully understand this trend.

However, the rate of increase was notably greater in the areas thought to be water absorption peaks, indicating that the drying process was causing reflectance to improve in those areas outside of the overall rise in reflectance seen throughout the test. This shows that the peaks were indeed water peaks and that this water could easily be removed, at least in part.



Figure 105. Reflectance data for Y₂O₃ sample 20Y-49 drying with nitrogen gas flow across its surface.

Test 4 – Drying Spectralon[®] v2

After seeing that both the Spectralon[®] and 20Y-49 had an overall upward shift in reflectance with every dataset, a longer duration test of drying Spectralon[®] was conducted to determine if and when the reflectance would eventually stabilize.

Procedure

- 1. Collect a baseline using NIST-traceable Spectralon[®].
- 2. Begin gas flow to dry the Spectralon[®].
- 3. Collect reflectance data for the Spectralon[®] immediately after beginning gas flow.
- 4. Continue to collect reflectance data for the Spectralon[®] periodically during 90 minutes of drying.
- 5. Collect a new baseline after 95 minutes of drying.
- 6. Collect another set of reflectance data after 100 minutes of drying, using the new baseline.
- 7. Turn off gas flow.

Results

Figure 106 shows reflectance data collected during this test. Results were similar to the first Spectralon[®] drying test early on, with the overall change decreasing over time. There was very little change between the datasets collected at 75 and 90 minutes. This is particularly apparent in Figure 107, which is zoomed in on one of the water peaks for this data.

Because the reflectance values for the Spectralon[®] appeared to have mostly stabilized, 95 minutes was chosen as the appropriate time to collect a new baseline for further testing.



Figure 106. Reflectance data for Spectralon® drying with nitrogen gas flow across its surface.



Figure 107. Reflectance data for Spectralon[®] drying with nitrogen gas flow across its surface – zoomed in on a water peak.

Figure 108 shows the last dataset collected, after 100 minutes of drying and using the new baseline. These data look typical of reflectance data for Spectralon[®], fluctuating around 100%, indicating that the sample collected after 100 minutes of drying was a match to the baseline collected after 95 minutes of drying.



Figure 108. Reflectance data for Spectralon® after 100 minutes of drying with nitrogen gas blowing across its surface, using a baseline of Spectralon® dried for 95 minutes.

Test 5 – Drying Y₂O₃ v2

Based on the continued changes in the Spectralon[®] over a longer timeframe, another test was run to observe changes in a rigid Y_2O_3 sample while drying it using the baseline collected from Spectralon[®] which had dried for 95 minutes. At the end of this test, data were also collected after turning off the gas flow to observe the sample reabsorbing water from the air.

Procedure

- 1. This test began immediately following Test 4, and using the baseline collected from Spectralon[®] that had been dried for 95 minutes.
- 2. Collect reflectance data for 20Y-49 prior to gas flow.
- 3. Turn on gas flow, drying the sample.
- 4. Collect reflectance data for 20Y-49 immediately after beginning gas flow, t = 0.
- 5. Continue to collect reflectance data for 20Y-49 periodically during 3 ½ hours of drying.
- 6. Turn off gas flow, allowing the sample to reabsorb water.
- 7. Collect reflectance data for 20Y-49 immediately after stopping gas flow, t = 0.
- 8. Continue to collect reflectance data for 20Y-49 every five minutes for 30 minutes, then again at 45 minutes while the sample sits, undisturbed, in ambient conditions.

Results

Figure 109 shows the reflectance data for the drying portion of this test. Figure 110 shows these data again, zoomed in on the water absorption peaks. In this test, there was almost no upward shift for each

dataset, outside of the water absorption peaks, as 20Y-49 dried. Starting with this sample much drier than it had been the previous day could have reduced the upward shift in reflectance outside of the water peaks in this dataset, in addition to any impact that the drier Spectralon[®] used for the baseline could have had.

There was a significant shift in the reflectance data collected before beginning gas flow to the one collected immediately after starting the gas flow, indicating that some water was removed immediately. There was also a significant shift between the datasets collected at 0 and 15 minutes of gas flow. After that, change was minimal between runs, suggesting that most of the water removed by this method was removed during the first 15 minutes.

After 3 ½ hours of drying, the water absorption peaks had changed very little since 30 minutes of drying, but they had not completely gone away. This suggests that not all of the water can be removed using this method of blowing air across the surface. Perhaps additional water remains inside of the sample, unaffected by the gas flow.



Figure 109. Reflectance data for Y₂O₃ sample 20Y-49 drying with nitrogen gas flow across its surface, using a baseline of Spectralon[®] that had dried for 95 minutes.



Figure 110. Reflectance data for Y₂O₃ sample 20Y-49 drying with nitrogen gas flow across its surface, using a baseline of Spectralon[®] that had dried for 95 minutes – zoomed in on a water absorption peak.

Figure 111 shows the reflectance data for the water reabsorption portion of this test. The original dataset, from before drying began, and the last dataset collected while nitrogen was still flowing, drying the sample (after 210 minutes of drying), are included for comparison. Figure 112 shows this same dataset, zoomed in on a water absorption peak.

There was little difference between those data and the data collected immediately after turning off the gas flow, indicating that little water returned immediately. After five minutes, roughly half of the water had returned. Most of the remaining water returned gradually over about 25 minutes, when reabsorption slowed significantly.

After 45 minutes, however, the water peaks still had not returned to their pre-drying levels. This could indicate that the reabsorption process is very slow, but it is also possible that some of the removed water will not return.



Figure 111. Reflectance data for Y₂O₃ sample 20Y-49 as it reabsorbs water under ambient conditions, after 210 minutes of drying with nitrogen gas flow across its surface.



Figure 112. Reflectance data for Y_2O_3 sample 20Y-49 reabsorbing water under ambient conditions, after 210 minutes of drying with nitrogen gas flow across its surface – zoomed in on a water absorption peak.

Test 6 – Drying a new Y₂O₃ Sample

The previous test indicated that some water remained inside of the sample, even after a lengthy period of drying by blowing air across the surface. Because the sample used in that testing, 20Y-49, had previously had additional drops of water added to it, the last test was repeated using a new Y_2O_3 sample, 20Y-50. This allowed the drying effects to be observed on a sample that had not potentially been impacted by the presence of additional water.

Procedure

- 1. Collect a baseline using NIST-traceable Spectralon[®].
- 2. Collect reflectance data for the Spectralon[®] prior to gas flow.
- 3. Begin gas flow to dry the Spectralon[®].
- 4. Collect reflectance data for the Spectralon[®] immediately after beginning gas flow, t = 0.
- 5. Collect reflectance data for the Spectralon[®] after 90 minutes of drying.
- 6. Collect a new baseline after 95 minutes of drying.
- 7. Collect another set of reflectance data for the Spectralon[®] after 100 minutes of drying, using the new baseline.
- 8. Turn off gas flow.
- 9. Collect reflectance data for the sample 20Y-50 prior to drying.
- 10. Begin gas flow, drying the sample.
- 11. Collect reflectance data for 20Y-50 immediately after beginning gas flow, t = 0.
- 12. Continue to collect reflectance data for 20Y-50 after 15 and 30 minutes of drying.
- 13. Turn off gas flow.

Figure 113 shows the reflectance data collected for Spectralon[®] during the first 90 minutes of drying. An inverted water peak not present in the data collected before drying appears in the data collected immediately after drying began, indicating immediate water removal on the Spectralon[®]. There was an unexplained downward shift across all wavelengths for the data collected after 90 minutes of drying, but the peak heights were similar.



Figure 113. Reflectance data for Spectralon[®] dried for 90 minutes by blowing nitrogen gas across its *surface.*

Figure 114 shows the reflectance data collected for 20Y-50 during this test. Figure 115 shows the same data, zoomed in on a water absorption peak. Again, using the longer drying time for Spectralon[®] before collecting the baseline, the reflectance remains nearly constant at wavelengths not affected by water absorption.

As expected, there was a significant change in the magnitude of the water absorption peaks between the data collected before drying began and the data collected immediately after starting the gas flow. There was a small change from the data collected at the start of gas flow to the data collected after 15 minutes of drying, but almost no change between 15 and 30 minutes of drying. Because of that, coupled with the minimal continued change over time in the previous test, this test was stopped after 30 minutes.

As with the prior test, the water absorption peaks were not completely eliminated by blowing air across the surface. This indicates that the water inside of the samples, which was unaffected when drying in this manner, was not the result of the water drops added to 20Y-49 in the initial test, but is a part of all rigid Y_2O_3 samples.



Figure 114. Reflectance data for Y_2O_3 sample 20Y-50 drying with nitrogen gas blowing across its surface.



Figure 115. Reflectance data for Y_2O_3 sample 20Y-50 drying with nitrogen gas blowing across its surface - zoomed in on a water absorption peak.

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