Introduction

Under contract to NASA, NanoLab developed several coating processes to produce very high absorbance optical blacks for stray light suppression. The original Singularity Black required a 300˚C bakeout and was replaced with the Low Temperature (LT) version to enable its use on more substrates. NanoLab offers three coatings to meet the needs of optical engineers:

- IM-1.6- designed for absorbance of light at glass to coating interfaces
- Singularity Black LT Aero (SB-LT)-for absorbance at air to coating interfaces.
- Singularity Black LT-LVOC Aero (SB-LT-LVOC)-low volatile organic solvent version.

Singularity Black Overview

Singularity Black paints are carbon nanotube filled, solvent-based paints that have world class performance in the visible, NIR and IR (typically below 1% reflectance in the visible), exceeding the optical properties of other commercially available products like Aeroglaze Z307 and Martin Black. Singularity Black's grazing angle performance is unmatched. Singularity Black paints are field repairable, should a coating become scratched or damaged.

Singularity Black comes in two formulations, SB-LT-Aero and SB-LT-LVOC Aero. The SB-LT-LVOC Aero has the same fillers and binders as standard SB- LT Aero, but the solvents have been replaced with a more environmentally friendly system. The SB-LT-LVOC formulation is slower to evaporate, but otherwise retains the same properties as the SB-LT formulation. The slower evaporation rate requires an adjustment to the spraying technique, but the SB-LT-LVOC has a longer shelf life.

Coating Services and Contact Information

In addition to selling the coatings, NanoLab provides coating services for baffles, telescope components, and other optical parts. Parts are handled in a class 100 clean space to prevent particulate contamination during the coating process. All work is done domestically, by US citizens in an ITAR compliant facility. Email sales@nano-lab.com for product quotes and coating services. Automated sprayers enable high throughput coating.

Singularity™ Black Training & Packaging

For those interested in painting components, we offer a Singularity School where students will learn safety protocol, and application techniques to make the deepest blacks possible. These courses can be held at NanoLab, or at your facility. We sell Singularity Black in four sizes: 20 ml, 250 ml, 500 ml & 1000 ml.

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Singularity Black Application

Air brush spraying is the recommended application technique. Manual brush application will not result in the same structure nor the same level of blackness.

Equipment

The equipment on the list below is in use at the NanoLab facility. It is strongly recommended to use similar equipment for paint application and clean part handling. Additionally, cleaning supplies (isopropyl alcohol, acetone, de-ionized water), swabs and other tools are required to clean and maintain the spray guns.

Ultrasonic bath	Solvents and detergents for part cleaning as needed
Spray booth	ventilation, 100 linear fpm minimum
Airbrush	Iwata Eclipse, or HP-TH2,
Air Supply	Clean, dry air supply, 1-2 CFM at 40PSI
PPE	Gloves, solvent vapor/particulate masks
Oven	130˚C +/- 5C with 10-minute timer
Environment	Class 100 clean bench for part handling before and after coating, curing and while packaging.

Material substrates suitable for Singularity Black Paints

Substrates and spray guns for SB-LT should be resistant to chemical attack from solvents such as methyl ethyl ketone and tetrahydrofuran. The SB-LT-LVOC formulation can be applied to a wider range of polymeric materials, but substrates should be resistant to water, alcohols, and alkanes. All surfaces should be free from loose particulate and organic residues that may reduce paint and primer adhesion. We have produced adherent, highly black coatings on the following materials:

Metals			
Aluminum alloys 2024, 6061	Chromate conversion coated aluminum (DTL Type 2, Class 1A)	Anodized aluminum (Type II, clear or PAA)	Mild & stainless steels
Nickel	Molybdenum	Copper	Titanium
Plastics			
Ultem 1010 (FDM)	Antero 840CN03 (carbon fiber filled PEKK) (FDM)	Antero 800NA (FDM)	Somos PerFORM (SLA)
Oxides			
Glass	Sapphire	Magnesium oxide	Aluminum oxide

Primer

For all metals, oxides, and some plastics, we recommend a silane pretreatment which will enhance the adhesive strength of the paint-substrate interface. Parts can be either sprayed (with the above airbrushes) or dipped in a Silquest™ A-187 (sourced from Momentive Performance Materials) epoxy functional silane, mixed at 1 wt.% in 85% ethanol/15% water.

After drying, this pretreatment leaves only a molecular layer thickness which does not measurably change the part dimensions. Silane treated parts are then baked to 130°C for 10 minutes to create a durable silane linkage on the surface. The silane treatment increases the bond strength of the coating-substrate interface beyond the cohesive strength of the coating. In practice, silane pretreated and Singularity coated articles rarely expose the substrate when scratched.

Top-coating with Singularity Black products

Mixing

SB-LT and SB-LT-LVOC are delivered in a ready to spray condition as a single component paint, No dilution or addition of second part is required. Paint should be agitated manually, mechanically or ultrasonically before use. Use lids to reduce evaporation of the paint in the sprayer-cup.

Coating

The proper gun to substrate distance for the Iwata Eclipse airbrush is ~10 cm (4"). After 8-10 coats, the coverage is ~0.65 to 1 ml/cm^2, which results in a 20-40 micron thick coating that performs well in the IR and visible. Thinner coats may perform adequately in the visible. Application of Singularity Black paints in multiple, thin layers is recommended, ensuring that at no time does the coating appear wet on the surface The solvent will start to evaporate in ambient conditions, causing the part to cool and the coating to appear dry. As more coating is applied, the rate of solvent evaporation will slow and a longer delay, up to 20 seconds of flash off time in between coats may be required. The SB-LT-LVOC formulation may require 30 seconds between coats to account for its slower evaporation rate.

Cure

After spray application, a heating step is required to crosslink the coating. Once crosslinked, it is a 'low touch' surface, tough enough for gentle handling. The curing of both types of Singularity Black paint require that the part see 130°C in air for 10 minutes to crosslink the binders and create a durable coating. Large parts may require instrumentation to monitor the temperature.

Analytical & Quality Control

To confirm that the coating has achieved its potential for sub 1% reflectance in the visible, NanoLab coats a witness sample with each coating job. That witness sample is typically a 2" square sheet of 6061 aluminum, which receives the same pretreatment and number of coats as the parts. That sample is baked alongside the parts, and then analyzed for total hemispherical reflectance. A UV-Vis Spectrometer equipped with an integrating sphere is used to measure the reflectance of the witness sample across the visible range.

If dry film thickness must also be measured, a mild steel witness sample of similar dimensions is also coated. We use a magnetic thickness gauge such as the Elcometer 211, as the coating is conductive, making eddy current measurements are inaccurate.

Handling & Packaging of coated parts

Packaging solutions should be designed to minimize contact with any coated surfaces. Standard practice is to mount parts on a plate with a standoff and then bag and seal both the plate and coated part to exclude dust. Packaging and handling at the NanoLab facility is conducted within a Class 100 clean bench.

Safety, Storage and Shelf Life of Singularity Black products

The shelf life of SB-LT is six (6) months from date of shipment when stored in the original, unopened container at temperatures between 4-25°C.(39-77°F) Containers will be marked with

an expiration date. Store indoors away from heat, sparks, and open flames. The pot life of SB-LT is at least three hours at 25°C (77°F) and 50% relative humidity, when protected from evaporation. Review the SDS for safety information on Singularity Black products.

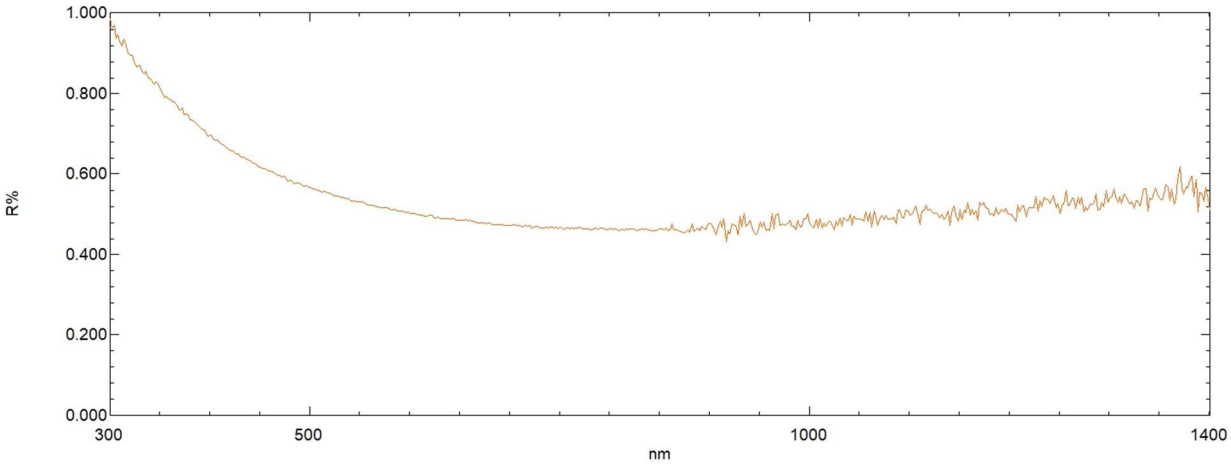
SB-LT-LVOC has a longer shelf life of twelve (12) months from date of shipment when stored in the original, unopened container at temperatures between 4-25°C.(39-77°F). The viscosity of fresh SB-LT-LVOC should be less than 100 cP, (Zahn #4 cup, <11 seconds). Expired paint that has become overly concentrated is difficult to spray and has a viscosity greater than 180 cP (Zahn #4 cup, >17 seconds).

Singularity Black, Properties of coated articles

Singularity Black Optical & IR performance

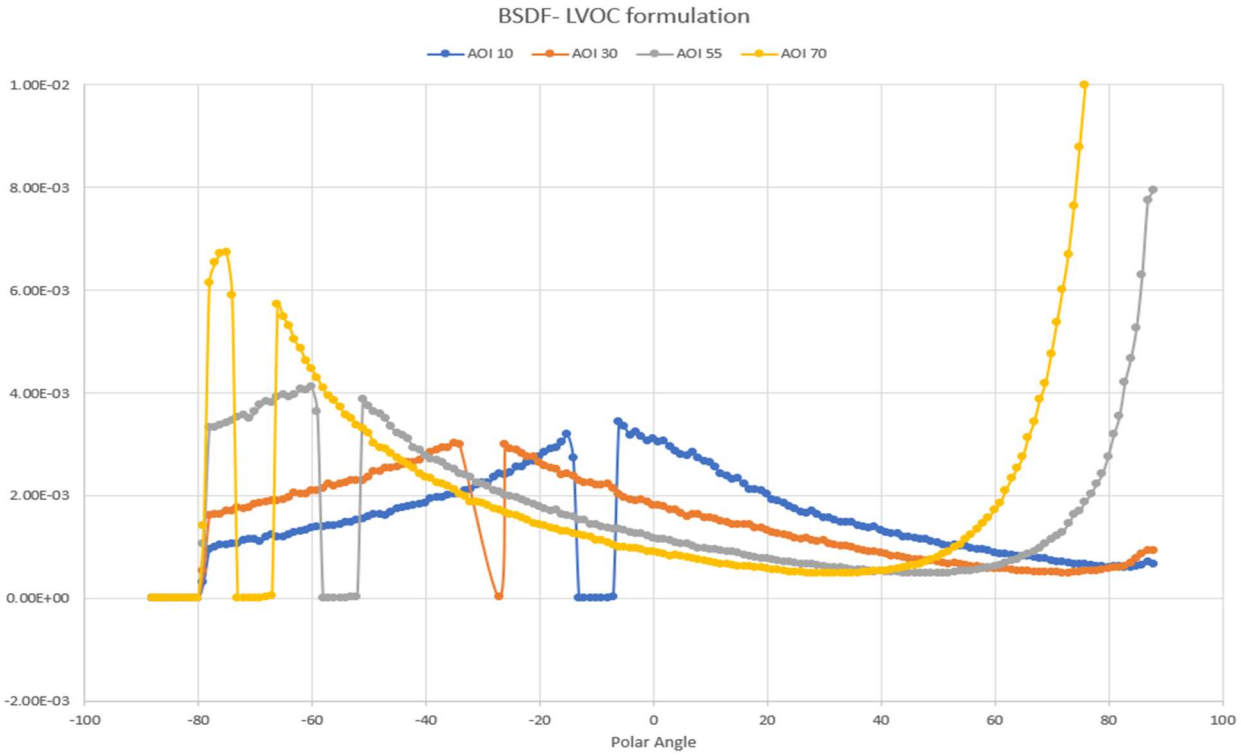
Total Hemispherical Reflectance (THR)

Singularity Black samples were measured at NanoLab, using our research grade UV-Vis-NIR spectrometer equipped with an integrating sphere, and measured referenced to a Spectralon white sample. We guarantee parts sprayed at NanoLab to achieve sub 1% THR in the visible.



BSDF

Bidirectional Scatter Distribution Function measurements were taken in 2022 at The Scatterworks, using their Complete Angle Scatter Instrument (CASI) with the source at incidence angles of 10, 30, 55 & 70 degrees. The detector scan radius covers the range +/- 90 degrees to the surface normal. The test wavelength was 1.06 micron.



Outgas Testing

Aluminum foil samples were coated with SB-LT for ASTM E-595-07 outgassing tests. Before testing, each foil was baked for 1 hour at 150°C. After testing, there was no visually detectable material on the collection plate.

ASTM E595 Outgassing test				
Date	**Notes**	**TML**	**WVR**	**CVCM**
7/16/2019	SB-LT, Lot T0605-3 on Al foil, Post cure 1 hour @150C	4.15	0.16	0.08

Outgassing tests on paint chip samples of SB-LT-LVOC were performed in April, 2021 by Aerospace & Advanced Composites GmbH, Austria, according ECSS-Q-70-02, The test is akin to ASTM E595 and was conducted at 125°C and 8 x10^{-7} Torr. Prior to test, the samples were post-cured at 150°C for 1 hour. The coating passes acceptance limits for ECSS-Q-70-02, which require the mean values be: RML < 1.0 % and CVCM < 0.10 %.

Material (See also material identification cards.)	TML		RML		WVR		CVCM		Acceptance limits achieved:
	Mean [%]	StdDev [%]	Mean [%]	StdDev [%]	Mean [%]	StdDev [%]	Mean [%]	StdDev [%]	
nanoLab TM 12-040	**0,34**	0,01	**0,33**	0,01	**0,01**	0,00	**0,02**	0,00	YES

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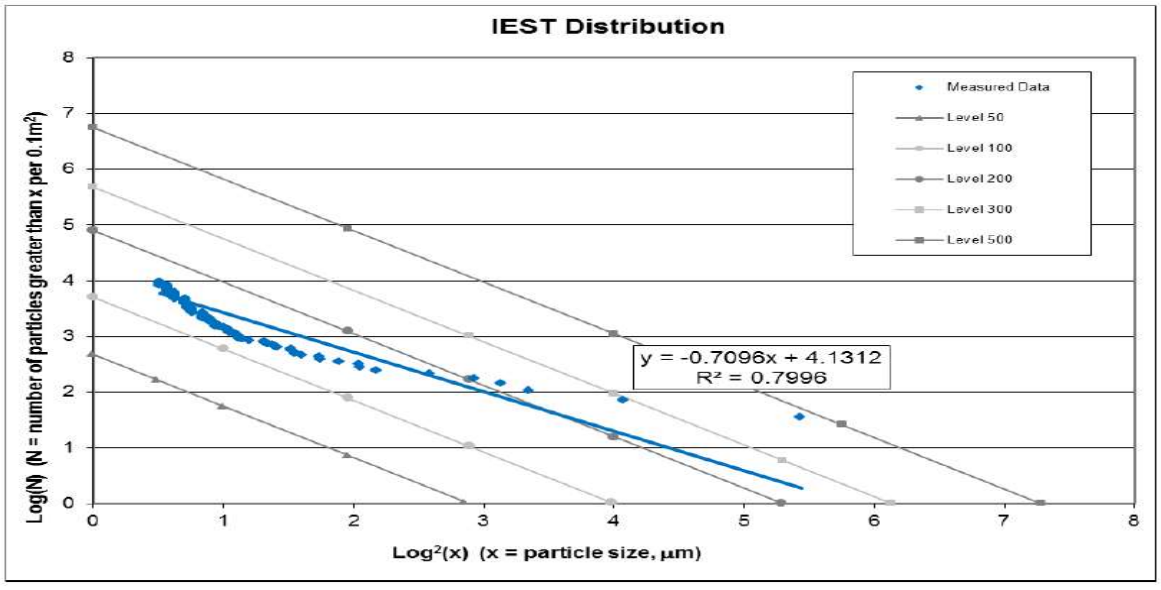
Cleanliness

The coatings are cleanable using gaseous nitrogen at low to medium pressure (5 to 50 psi). The process removes stray fibers and dust, but not the coating. Once cleaned, the coatings were measured with a Clemex particle counter. Many optical instruments have a requirement of Level 400 per Institute of Environmental Sciences and Technology (IEST) STD-CC1246E. Assemblies with slits or pinholes require Level 200. Painted hardware typically has a particle level higher than 500. The IEST equivalent for the Singularity sample was 127.

Total Particles	266	**Total Area Counted (μm^2)**	2.813E+09
Background Particles	0	**Area of Particles (μm^2)**	1.848E+04
		Percent Area Covered (PAC)	6.571E-04
Particles Analyzed (total - background)	266	**Background PAC**	0.000E+00
		PAC (total - background)	6.571E-04
Particles Analyzed (total - background) per $0.1m^2$	9457	**PAC rate**	
		Equivalent IEST Level*	**127**

Particle Size µm	ALLOWABLE PARTICLES per $0.1m^2$ BY IEST CONTAMINATION LEVEL					Number of Others per $0.1m^2$	Number of Fibers per $0.1m^2$	Total Particles per $0.1m^2$
	100	200	300	400	500			
>25	78.4	1230	7450	28800	86300	284	36	320
>50	10.7	169	1020	3950	11800	142	36	178
>100	1	15.8	95	367	1090	36	36	71
>200		1	6	23.3	69.6	0	36	36
>300			1	3.9	11.6	0	0	0
>400				1	3	0	0	0
>500					1	0	0	0



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Vibration

The Singularity coated panels successfully passed vibration testing. The GEVS vibration level is an input of 14.1 Grms that comes from the "General Environmental and Verification Specification for STS and ELV Payloads, Subsystems and Components", GEVS-SE, June 1996, Table 2.4-4. The BPL test is from a Ball Aerospace Structural Guideline and the vibe levels (also called Power Spectral Density or PSD) are high to account for design cases where responses cannot be predicted. It includes a combination of vibe levels that account for acoustic vibration, random vibration, and quasi-static loading. This level is higher than most flight applications. No visible particles were removed during these tests. Some particles were generated by the vibration testing and characterized using the Clemex particle analyzer.

Vibe Level	GEVS				BPL			
Test Order	1	2	3	4	5	6	7	8
Sample	129-6	140-5	129-6	140-5	129-6	140-5	129-6	140-5
Axis	X	X	Z	Z	Z	Z	X	X
PC Test	15-0705	15-0708	15-0706	15-0707	15-0704	15-0703	15-0701	15-0702
micrometers	Particles per 0.1 m2							
>25	1201	426	543	659	504	465	1783	1473
>50	271	155	155	116	116	155	426	426
>100	0	116	0	78	0	116	310	194
>250	0	39	0	78	0	78	78	78
>500	0	0	0	78	0	78	78	39
Percent Area Covered (PAC)	2.86E-03	4.19E-03	1.19E-03	1.36E-02	1.19E-03	1.46E-02	2.53E-02	1.83E-02
IEST-CC-1246 Level	**100**	**200**	**148**	**262**	**248**	**266**	**300**	**280**
Comments	Dark	Dark	Mostly dark	Mostly black + 2 Blue	Mostly dark	Dark	None	Mostly black

Particle fallout levels during vibration testing were between IEST Levels 100 and 300, which are lower than what is observed for Martin Black and other black paints.

Thermal Stability Data

Samples were thermal cycled three times between -60°C and 85°C at a ramp rate of 10°C/minute, with 30-minute dwells at the extremes, in a nitrogen atmosphere. No damage or loss of particulate was noted. Also, thermal vacuum conditions, $<1e^{-6}$ torr at 100°C, did not affect the coating.

The safe upper operational limit for Singularity Black coating is 350°C in air, 400°C in vacuum, and safe lower operational limits for the coating are -200°C in air or vacuum.

The autogenous autoignition temperature for the coating is 600°C in air. Combustion products may include CO, CO_2.

Other

Conductivity	~150 ohm per square.
Emissivity	0.86
Absorptivity	0.99
UV Degradation	Expected high resistance [1]
Atomic Oxygen	Yet untested, expected high resistance [2]
Ionizing Radiation	Untested, expected improvement over unfilled polymers [3]
Wetting	Hydrophobic.
Wet odor	Ethereal
Dried odor	None

Estimates for Ultraviolet and Atomic Oxygen degradation are based on literature review for nanotube loaded polymeric composites.

1. "Effects of UV degradation on surface hydrophobicity, crack, and thickness of MWCNT-based nanocomposite coatings," R. Asmatulu, G.A. Mahmud, et al. Progress in Organic Coatings, Volume 72, Issue 3, November 2011, Pages 553-561 https://doi.org/10.1016/j.porgcoat.2011.06.015
2. "Reactive Molecular Simulation of the Damage Mitigation Efficacy of POSS-, Graphene-, and Carbon Nanotube-Loaded Polyimide Coatings Exposed to Atomic Oxygen Bombardment," F.Rahmani, S.Nouranian, et al. ACS Appl. Mater. Interfaces 2017, 9, 14, 12802–12811, https://doi.org/10.1021/acsami.7b02032
3. "Ionizing Radiation Effects on Interfaces in Carbon Nanotube-Polymer Composites", J.. Harmon, P.. Muisener, M. Meyyappan, A. Cassell, MRS Online Proceedings Library (OPL) , Volume 697: Symposium P – Surface Engineering 2001--Fundamentals and Applications , P9.7 https://doi.org/10.1557/PROC-697-P9.71