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Evaluation of Select High Temperature Coatings

by Thomas A Considine, John V Kelley, David Orsini, and Tyler Sexton

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<p>Touch-up painting is performed in the field to restore the authorized coatings applied on US Army and other US Military ground equipment platforms. However, due to the requirements for cure of the specified high temperature coatings for steel substrates (MIL-PRF-14105) they cannot be applied in a field setting. This precludes their use by Soldiers during touch-up painting operations (performed in accordance with Technical Manual 43-0242) and often results in improper cure of these coatings, even when applied at a Logistics Readiness Center, Materiel Maintenance Division, and other organic re-paint facilities within the Army, due to a lack of facilities to bake these parts. This results in either touch-up painting not being performed or premature failure of these coatings on exhaust systems and other components leading to corrosion of the underlying steel substrate. There is a need for touch-up coating products that can be used in high temperature applications. This report evaluates a selection of commercially available high temperature coatings for touch-up applications.</p>					
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1. Introduction

The standard Chemical Agent Resistant Coating (CARC) system, used across most US Military ground equipment platforms, benefits from having several touch-up applications proven out and in use at depots, installations, and even by troops in the field. More specialized components bearing the CARC system, such as those requiring high heat coatings, lack options for touch-up due to their particular nature. The coatings qualified to MIL-PRF-14105,¹ *Paint, Heat-Resisting (For Steel Surfaces)*, require curing at an elevated temperature in an oven or similar equipment. This is not feasible at the field level and is both labor and energy intensive at facilities that may have the proper equipment to provide a cure to parts removed for touch-up applications. This may result in the use of an incorrect coating, or the appropriate coatings being improperly applied. The purpose of this study is to evaluate a range of commercial off-the-shelf (COTS) high temperature coatings that can be used to provide a viable touch-up coating option even if they do not fully meet the current requirements of MIL-PRF-14105. A series of adhesion, corrosion, and thermal cycling tests were employed to identify potential coating candidates.

2. Experimental Procedure

Sample flat test coupons of American Society for Testing and Materials (ASTM) International A1008/1010² steel ($3 \times 6 \times 1/8$ inches), were abrasive-blasted with 60-grit aluminum oxide to achieve a uniform surface profile of approximately 1.5 mil to conform with the stated profile depth required by MIL-PRF-14105 (Table 1). A second, smaller set of panels (4×4 inches) were created for abrasion testing from the same material.

Table 1 Coating test matrix

Manufacturer	Coating	Mandrel bend	Cross hatch	Salt spray	Thermal + salt spray
Cerakote	C-7600	1	1	3	3
	V-169	1	1	3	3
	C-192	1	1	3	3
DuraCoat	DuraHeat 2.0	1	1	3	3

The high heat coatings were applied in accordance with (IAW) manufacturer recommendations by US Army Combat Capabilities Development Command (DEVCOM) Army Research Laboratory (ARL) personnel. For Cerakote C-7600 and V-169, panels were provided by the manufacturer.

The thermal cycling was carried out using a modified version of MIL-PRF-14105 in order to obtain more data points and assess performance over more temperature intervals. The thermal cycling and testing schedule follows:

- 1) All panels in oven.
- 2) Heat to 400 °F for 8 h, turn off oven and cool to room temperature over 16 h.
- 3) Heat to 650 °F for 8 h, turn off oven and cool to room temperature over 16 h.
- 4) Heat to 800 °F for 8 h, turn off oven and cool to room temperature over 16 h.
 - a. Remove two panels for corrosion test.
- 5) Heat to 1200 °F for 8 h, turn off oven and cool to room temperature over 16 h.
 - a. Remove two panels for corrosion test.
- 6) Heat to 1400 °F for 8 h, turn off oven and cool to room temperature over 16 h.
 - a. Remove two panels for corrosion tests.
- 7) Conduct corrosion tests:
 - a. Expose:
 - i. Two panels (as received, no thermal cycling)
 - ii. Two panels after 800 °F
 - iii. Two panels after 1200 °F
 - iv. Two panels after 1400 °F

2.1 Mandrel Bend

Mandrel bend testing was performed IAW ASTM D522, Method B³ to evaluate the film layers' resistance to cracking. Each panel was centered upon and bent around a 0.25-inch mandrel at as close to a uniform velocity as possible, then inspected for cracks along the stressed area of the bent surface. Any cracking or distention of the coating was considered a failure.

2.2 Adhesion

Cross-hatch adhesion testing was performed IAW ASTM D3359, Method B.⁴ A cross-hatch adhesion test kit with multi-tooth cutter blade was used to make two intersecting perpendicular sets of six parallel cuts with 2-mm spacing through the film of the coating into the substrate. This intersecting set of 6×6 grids was repeated nine times across the panel. After scribing, the panels were brushed to remove any coating debris prior to testing. A full lap of an approved tape per TT-C-490⁵ was removed prior to testing. Tape was laid down across each scribed grid and smoothed into place using an adhesive test tool roller and left to set for 60 s. The tape was removed by grasping the free end and pulling the tape back along itself 180° . Each grid was rated using the classification guide in ASTM D3359, Method B (Fig. 1).





Surface of cross-cut area from which flaking has occurred. (Example for 6 parallel cuts).	None					Greater than 65%
Classification	5	4	3	2	1	0

Fig. 1 Cross-hatch adhesion rating scale

2.3 Taber Abrasion

Abrasion testing was performed IAW ASTM D4060⁶ to evaluate the resistance of coatings to rubbing abrasion. A Taber 5135 Rotary Abrasion Table (Fig. 2) was used to perform testing. Samples were weighed on a precision balance and values recorded prior to being abraded using CS-17 wheels and a 1000-g counterweight at 60 rpm. Rub-wear action of the Abraser is done by the contact of the test panel turning on a vertical axis, against the two rotating abrading wheels. One wheel rubs outward and the other inward resulting in a cross-hatch pattern. Test panels were evaluated for weight loss after breakthrough of the coating, the approximate cycle of which was recorded.

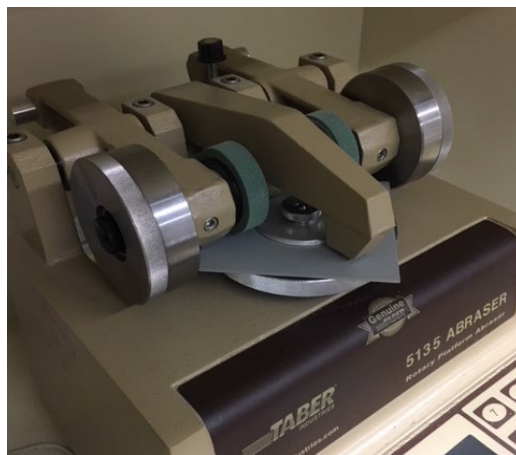


Fig. 2 Taber abrasion test setup

2.4 Corrosion Testing

The final method of testing was neutral salt fog testing over thermally cycled panels. Panels of each product were heated to 800 °F for 8 h, then allowed to cool to room temperature for 16 h. Two panels from each set were removed at this point, and the remaining panels were heated to 1200 °F. The cooling process was repeated, and two additional panels were removed before heating the remaining two panels to 1400 °F. Two unheated panels were reserved from thermal cycling to have a baseline unheated set of panels for each product. The panels were X-scribed IAW ARL procedure⁷ using a tungsten-carbide scribe to etch a line through the coating and into the substrate. Panels were exposed IAW ASTM B117⁸ in an Autotechnology Standard Salt Fog Chamber (Model 22) for 100 h (Fig. 3), removed from testing, rinsed in deionized water, and allowed to dry. Panels were photographed upon removal. Delaminated coating and loose corrosion products were removed using a 2-inch putty knife, then submerged in an Alconox solution to remove additional corrosion products, without removing any additional coating. Panels were given a final rinse, allowed to dry, and scanned using a flatbed scanner.



Fig. 3 Salt fog chambers

3. Results

3.1 Mandrel Bend

Mandrel bend testing was performed IAW ASTM D522. The pass/fail criteria for mandrel bend testing is no cracking or other degradation of the coating along the bend. All products tested passed mandrel bend without issue. One panel appeared to have a bit of a 0.25-inch-long marring along the fold line; however, on closer inspection it was determined that this was a superficial blemish on the coating. Photographs of each panel can be seen in Figs. 4–7.



Fig. 4 Cerakote C-192 following mandrel bend



Fig. 5 Cerakote C7600 following mandrel bend



Fig. 6 Cerakote V-169 following mandrel bend



Fig. 7 DuraCoat DuraHeat 2.0 following mandrel bend

3.2 Adhesion

Cross-hatch adhesion testing was performed IAW ASTM D3359, Method B. A new, unexpired roll of 3M 250 Flatback Masking Tape was used for this test. A full lap of tape was removed prior to testing. A cross-hatch test tool was used to scribe an intersecting 6×6 grid of lines at 1-mm intervals through the coating to the substrate. This was done as many times across each panel as surface area allowed, yielding 8–9 cross-hatch tests per panel depending on panel size. Every cross hatch on every product was rated at 5. A rating of greater than or equal to 4 is required by all CARC specifications as well as MIL-PRF-14105—meaning that every product met the requirements to pass cross-hatch adhesion testing. Images of each panel can be seen in Figs. 8–11.



Fig. 8 Cerakote C-192 cross-hatch adhesion results

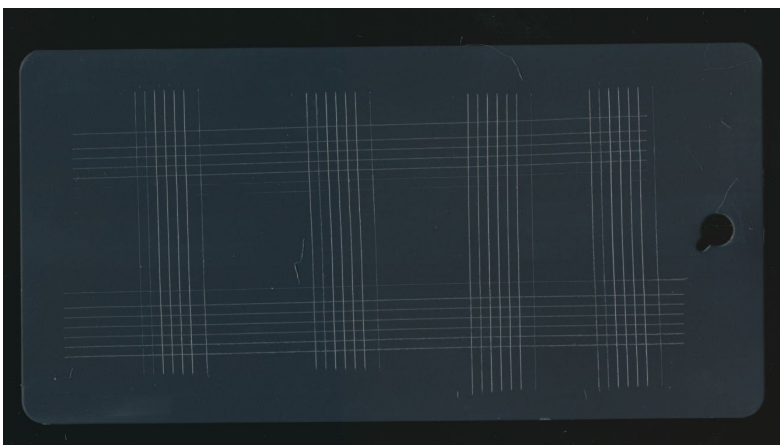


Fig. 9 Cerakote C7600 cross-hatch adhesion results

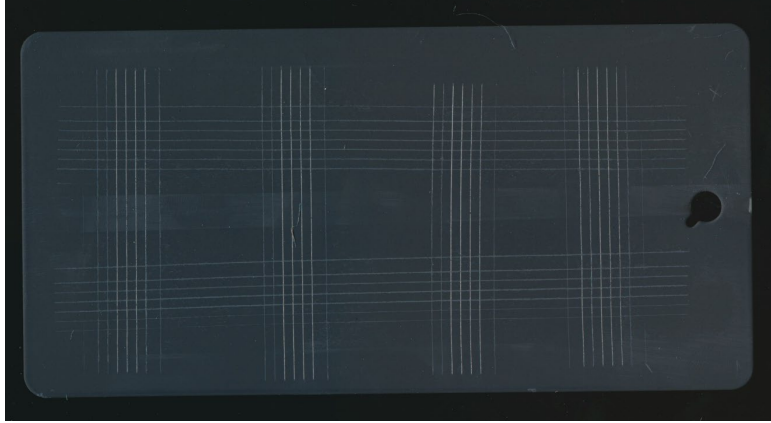


Fig. 10 Cerakote V-169 cross-hatch adhesion results

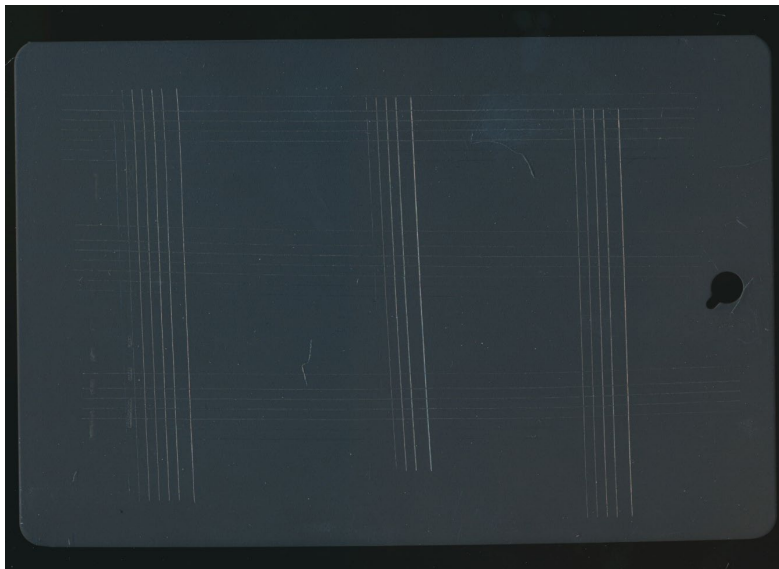


Fig. 11 DuraCoat DuraHeat 2.0 cross-hatch adhesion results

3.3 Taber Abrasion

Panels were tested IAW ASTM D4060 for abrasion resistance. Taber Abrader CS-17 wheels for coatings were used with a 1000-g arm load. Abrasion resistance is not a requirement in MIL-PRF-14105; therefore, this test was performed for comparative purposes only. Additionally, only two of the products were tested in this manner as the remaining two were not provided on the correct size panels. Of the two products tested, Cerakote C-192 lasted the longest under abrasion, taking approximately 2000 cycles before a coating breakthrough occurred, but was taken to 4000 cycles as full breakthrough had not occurred. Mass loss at initial

breakthrough was 0.043 g, and 0.069 g at full breakthrough. DuraCoat DuraHeat 2.0 abraded through considerably sooner, lasting about 250 cycles before full breakthrough. Mass loss at the time of breakthrough was 0.035 g. Images of each can be seen in Figs. 12 and 13.



Fig. 12 Cerakote C-192 following 4000 cycles of Taber abrasion

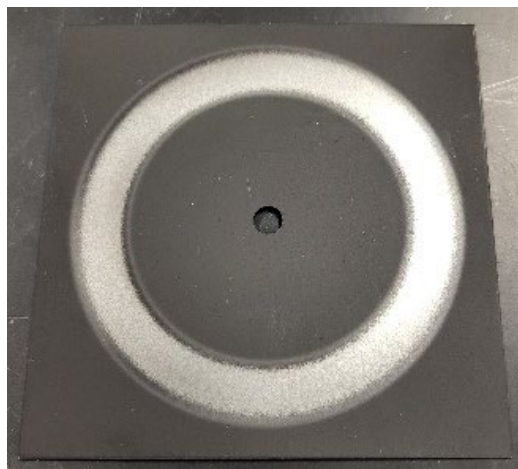


Fig. 13 DuraCoat DuraHeat 2.0 following 250 cycles of Taber abrasion

3.4 Corrosion Testing

Panels tested in ASTM B117 were rated IAW ASTM D1654⁹ for scribe corrosion and IAW ASTM D610¹⁰ for blistering in field. To conform with the specification requirements, scribe corrosion must be rated greater than or equal to 6, and blistering in field must be a grade 9 or higher with no more than five scattered blisters, each less than 1 mm in diameter. Samples of each product were tested in four different configurations: as-cured (air); and thermally cycled to 800, 1200, and

1400 °F. While typical corrosion testing is performed at just 800 °F, the additional elevated temperatures were also evaluated for corrosion resistance to see how the coating performs at extremes. These results and images are shown in Figs. 14–18.

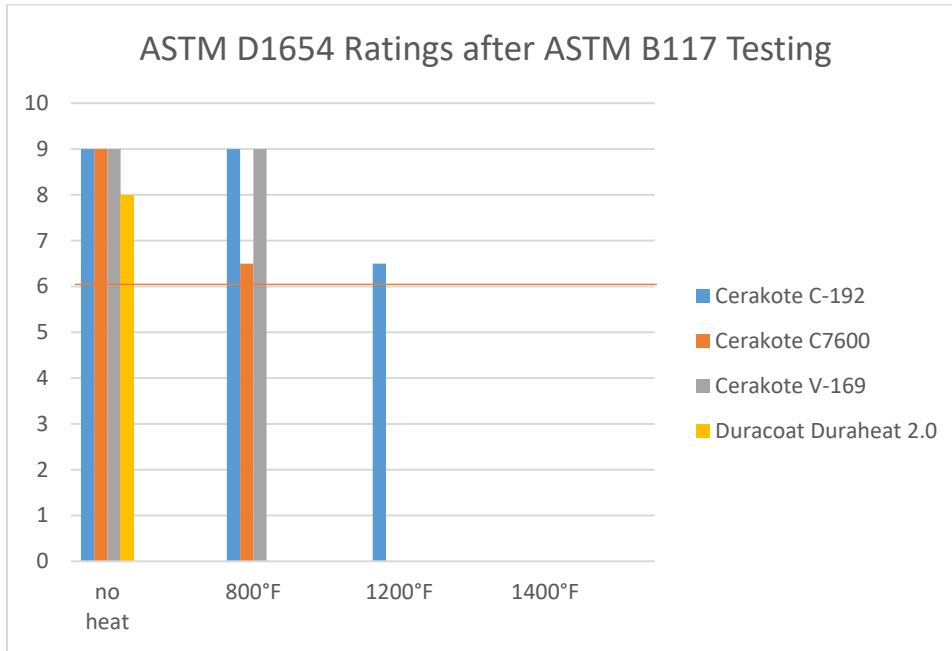


Fig. 14 ASTM D1654 ratings after ASTM B117 exposure

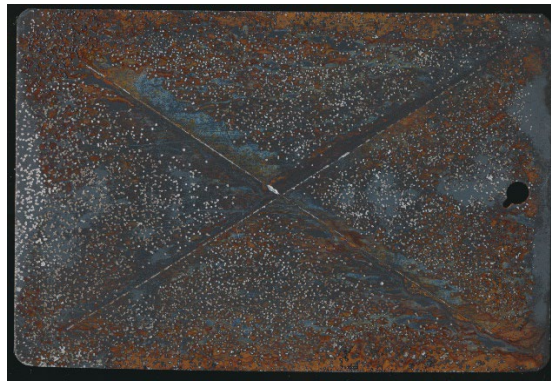


Fig. 15 Cerakote C-192 at 800 °F after ASTM B117 exposure



Fig. 16 Cerakote C7600 at 800 °F after ASTM B117 exposure



Fig. 17 Cerakote V-169 at 800 °F after ASTM B117 exposure



Fig. 18 DuraCoat DuraHeat 2.0 at 800 °F after ASTM B117 exposure

The elevated temperatures proved too demanding for several of the coatings tested, resulting in massive failures and making scribed ratings impossible. Only the “as-cured” set of panels all met the requirements to qualify as passing in salt fog testing, and two of the coatings continued to maintain corrosion resistance when thermally cycled to 800 °F as required by the specification. The Cerakote C7600 did not perform as effectively after the thermal cycling to 800 °F, but still met the specification requirement of greater than or equal to 6. Only the Cerakote C-192 would have been considered a pass at 1200 °F, while the remaining products lacked sufficient remaining coating to evaluate anything from the scribe. As a result, a secondary rating method was implemented: measuring the remaining coating as a percent of the surface area. To accomplish this, panels were scanned on a flatbed scanner and imported into GNU Image Manipulation Program (more commonly known as GIMP).¹¹ The images were converted to grayscale and the brightness and contrast were adjusted until the substrate and coating were clearly delineated in black and white. A histogram was then run to give an exact percentage of coating to substrate based on the black and white saturation values. These values are given in Fig. 19, and photographs are shown in Figs. 20–23.

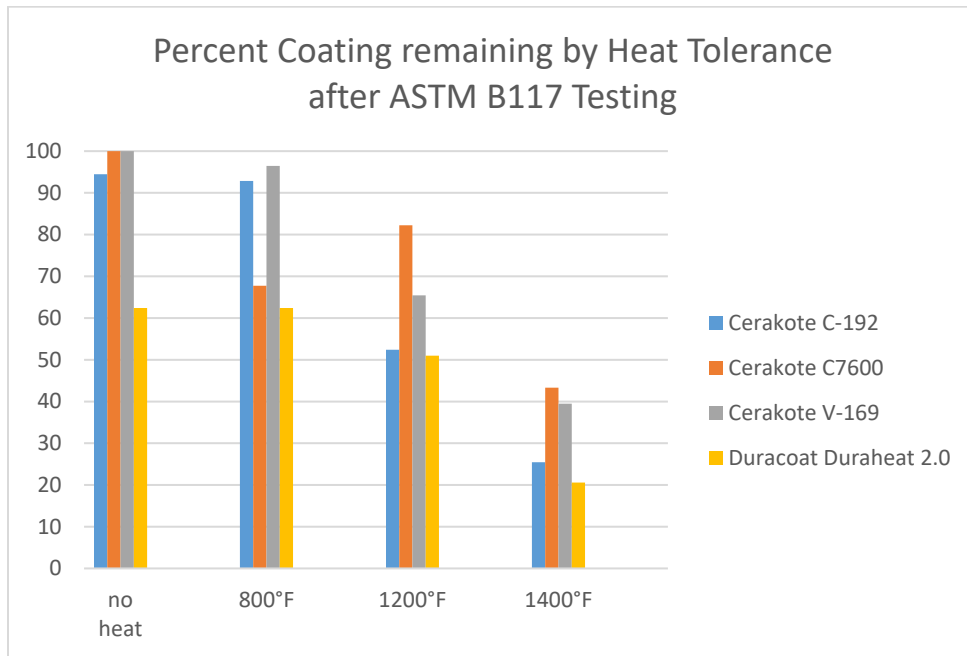


Fig. 19 Percentage of coating remaining following ASTM B117 exposure/heat exposure level

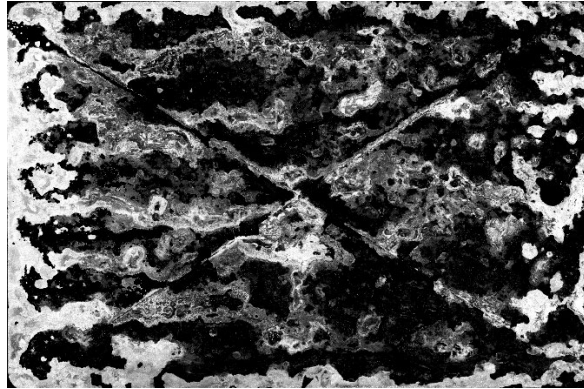


Fig. 20 Cerakote C-192 at 1200 °F, converted to grayscale for measurement

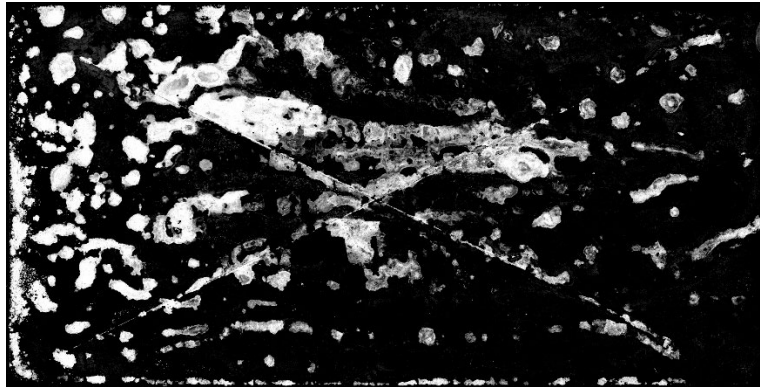


Fig. 21 Cerakote C7600 at 1200 °F, converted to grayscale for measurement

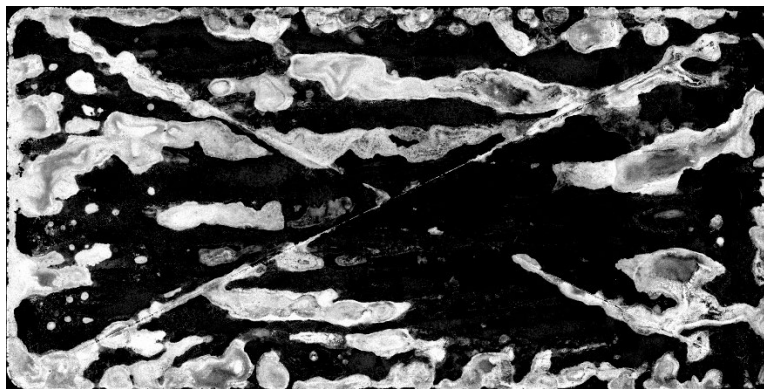


Fig. 22 Cerakote V-169 at 1200 °F, converted to grayscale for measurement

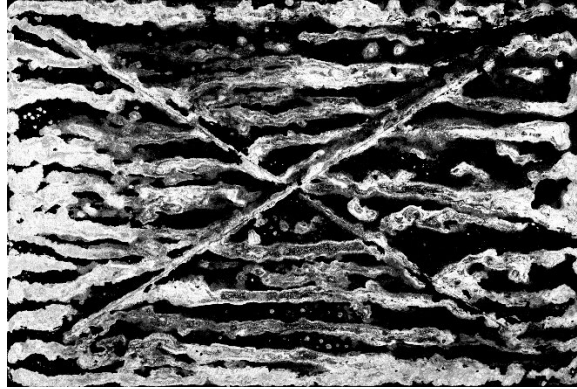


Fig. 23 DuraCoat DuraHeat 2.0 at 1200 °F, converted to grayscale for measurement

In general, a clear downward trend can be seen in coating remaining versus increase in temperature. There was one exception to this trend: the Cerakote C7600 retained less coating at 800 than 1200 °F. At 1400 °F, corrosion testing was not even considered, as an energetic coating anomaly was observed during the scribing process. As the scribe interacted with the surface of the test coupons, the coating began to pop off the panel entirely, and continued to do so after the scribe tool was removed. After conferring with the manufacturer, who had experienced this issue before on thin mild steels such as exhaust manifolds, it was determined that the coating itself was under tension from the sustained elevated temperatures. The scribing procedure provided relief, which caused the coating adjacent to the scribed areas to energetically delaminate.

4. Summary and Conclusion

It is worth emphasizing that this was a limited-scale study and coatings were not subjected to the full slate of qualification tests IAW MIL-PRF-14105. Rather, it was intended to be a quick evaluation of COTS products that could serve as touch-up coatings while awaiting a specification revision, given the absence of currently approved options. Adhesion, abrasion, thermal cycling, and corrosion resistance testing were selected based on their importance and brevity of testing. A full evaluation of products should be performed to ensure compatibility and provide data to support specification updates, potentially including touch-up parameters. In these tests, all of the coatings met and exceeded the requirements for adhesion testing through mandrel bend and cross-hatch tests. Abrasion resistance is not a requirement in the high heat specification and was performed only on two of the coatings due to panel size requirements versus what was provided by the manufacturer. Further, the abrasion resistance only captures the data for those two coatings; however, a baseline of a traditional high heat coating has not been performed for comparison.

Corrosion resistance on panels not subjected to thermal cycling was excellent; all coatings met and exceeded traditional corrosion requirements for CARC coatings on steel. The Cerakote products were also able to meet these requirements following thermal cycling to 800 °F, the temperature point after which corrosion is evaluated per MIL-PRF-14105. Cerakote C7600 was the only product that also met these corrosion requirements after exposure to 8 h of 1200 °F. Cycling up to 1400 °F proved too extreme for all coatings, causing a stress-based delamination of the coating when the scribing process acted as relief. As such, corrosion testing was not performed. This temperature is usually where quenching and accelerated weathering testing occurs, and scribing would not be performed, possibly obscuring similar results on additional products. Although this was only a limited scope study, the Cerakote products tested may be an acceptable option for high heat touch-up applications, provided service temperatures do not exceed 1200 °F. However, ARL recommends that full-scale testing be performed IAW MIL-PRF-14105. Furthermore, real-world operating temperature observations have not been consistent with the temperature range requirements in MIL-PRF-14105. This has led to calls for specification revision and the inclusion of additional types that conform to more realistic service conditions, which would warrant additional study and a re-evaluation of performance requirements.

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List of Symbols, Abbreviations, and Acronyms

ARL	Army Research Laboratory
ASTM	American Society for Testing and Materials
CARC	Chemical Agent Resistant Coating
COTS	commercial off-the-shelf
DEVCOM	US Army Combat Capabilities Development Command
GIMP	GNU Image Manipulation Program
IAW	in accordance with