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The Evaluation of Supercritical CO_2 Application of CARC

by Jeffrey Duncan, Donovan Harris,
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Weapons and Materials Research Directorate, ARL

Abstract

At the request of the U.S. Army Tank-automotive and Armaments Command (TACOM), the Coating Technologies Team of the U.S. Army Research Laboratory (ARL), Weapons and Materials Research Directorate (WMRD), evaluated a chemical-agent resistant coating (CARC) reformulated to be compatible with the supercritical CO_2 application process. Test panels were prepared by the Concurrent Technologies Corporation (CTC) and submitted to ARL's Experimental Products Program (EPP) for approval. Thus far, four different and separate formulations have been submitted, and all have failed, including the fourth and most recent submission prepared at the CTC on 12 November 1999. In each case, excessive voids and pinholes riddled the coating film applied with supercritical CO_2. To date, the use of supercritical CO_2 has not been successful in demonstrating its ability to apply CARCs free of voids or pinholes. This report summarizes the test program and the results through 24 November 1999.

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1. Background

For several years, TACOM has been interested in reducing the volatile organic compound (VOC) content of the U.S. Army's chemical-agent-resistant coating (CARC) by using supercritical CO_2 for spray application. This application process, marketed by Union Carbide under the patented UNICARB designation, requires reformulation of a standard coating for compatibility with supercritical CO_2 reduction. The U.S. Army Research Laboratory's (ARL) Coatings Technologies Team became involved because it is the activity responsible for CARC, including research and development (R&D), specification preparation, and the generation and maintenance of any associated qualified products list (QPL). The major concern was whether or not the supercritical CO_2 application process or the reformulation necessary would affect the performance of the applied coating. In addition, this change in composition itself is in direct conflict with the qualification process as governed by SD-6 [1], Provisions Governing Qualification (QPL).

Because the reformulated coatings do not meet specification composition requirements, the mechanism for evaluating them is the Experimental Products Program (EPP), set up by ARL to investigate performance-based alternatives to specification materials. It allows evaluation of products offering the Army benefits not mentioned by these specifications, such as improved performance or environmental acceptability. The set of tests used to evaluate the applied films was extracted from appropriate material specifications. In addition, photographic procedures were used to objectively demonstrate the film voids and pinholes in the supercritical CO_2 samples. Test panels for this program were prepared by the National Defense Center for Environmental Excellence (NDCEE) a part of the Concurrent Technologies Corporation, using a statement of work (SOW) developed mutually by the U.S. Army Tank-automotive and Armaments Command (TACOM) and ARL. The basic intent was to investigate the two potential problem areas previously mentioned: reformulation and application. The SOW called for test panels to be prepared using standard CARC applied both conventionally and via the supercritical CO_2 application process, along with reformulated CARC applied both conventionally and via the supercritical CO_2 application process. TACOM later added a fifth set of test panels, a reformulated lower-VOC CARC, applied via the supercritical CO_2 application process. The SOW called for a total of 25 panels to be prepared in each set, allowing for all of the EPP performance testing, and several extras in case of problems or a desire to repeat a particular test. The substrate of choice was 4-in × 12-in zinc phosphate-pretreated steel (TT-C-490, Type 1) with 1 mil of epoxy primer in accordance with MIL-P-53022 [2]. In addition, five phosphoric acid-etched panels for each set were prepared from thin steel (31 gauge) for flexibility testing. Hentzen Coatings, Inc. manufactured the topcoats. This report summarizes the test program and its results.

2. Test Procedures

As previously noted, since the panels were prepared by the NDCEE, the EPP testing focused on the performance of the cured dry films. Hentzen manufacturing codes and formulation data for the five sets of panels are listed in Table 1.

Upon delivery, the test panels were arbitrarily assigned to specific tests on the basis of the panel label. Tables 2 and 3 show the labels on the panels and the test for each.

Table 1. Coatings Descriptions

Set	Manufacturer's Code	Application Method	Description (no./gal VOC)
A	08605GUZ-GD	Supercritical CO_2	3.5
B	RLE14697GUZ	Supercritical CO_2	2.8
C	RLE15147GUZ	Supercritical CO_2	2.2
D	08605GUZ-GD	Conventional	3.5
E	RLE14697GUZ	Conventional	2.8 (thinned for spray)

Table 2. Panel Tests, 4-in × 12-in Size

Sequence No.	Panel Label					Test
	A	B	C	D	E	
1	1	1	9	1	1	Chemical-agent resistance
2	2	2	10	2	2	Gloss, color, IR, DS2 resistance, acid resistance
3	3	3	11	3	3	Recoat with MIL-C-46168 [3]
4	4	4	12	4	4	Recoat with MIL-C-53039 [4]
5	5	5	13	5	5	Recoat with MIL-P-64159 [5]
6	6	6	14	6	6	H_2O resistance, HC resistance
7	7	7	15	7	7	Lube oil resistance, hydraulic fluid resistance
8	8	8	16	8	8	Xenon-arc weathering
9	9	9	17	9	9	Xenon-arc weathering
10	10	10	18	10	10	QUV weathering
11	11	11	19	11	11	QUV weathering
12	17	12	20	12	12	Outdoor exposure
13	18	13	21	13	13	EMMAQUA
14	19	14	22	14	14	EMMAQUA
15	22	15	23	15	15	Wet/dry adhesion
16	23	16	24	16	16	(not assigned)
17	24	17	25	17	17	(not assigned)
18	25	18	26	18	18	ASTM B117 [6]
19	26	19	27	19	19	ASTM B117 [6]
20	27	20	28	20	20	ASTM B117 [6]
21	28	21	29	21	21	ASTM B117 [6]
22	29	22	30	22	22	GM 9540P [7]
23	30	23	31	23	23	GM 9540P [7]
24	31	24	32	24	24	GM 9540P [7]
25	32	39	39	25	25	GM 9540P [7]

Table 3. Panel Tests, 4-in × 6-in Size

Sequence No.	Panel Label					Test
	A	B	C	D	E	
1	35	25	1	33	31	1/4-in Mandrel Flexibility
2	36	26	2	34	32	1/4-in Mandrel Flexibility
3	37	27	3	35	33	1/4-in Mandrel Flexibility
4	38	28	4	36	34	1/4-in Mandrel Flexibility
5	39	29	5	37	35	1/4-in Mandrel Flexibility
6	—	30	—	—	—	1/4-in Mandrel Flexibility

3. Test Results

Prior to performing any tests, dry-film thickness measurements were performed on all of the submitted test panels. Results for the 4-in × 12-in panels are shown in Table 4, and the results for the 4-in × 6-in panels are shown in Table 5.

The measurements listed in Tables 4 and 5 are averages of eight readings taken per panel, and the format indicates the standard deviation as calculated by the Elcometer dry-film thickness gauge (i.e., 3.95/0.15 indicates an average film thickness of 3.95 ± 0.15 mil).

Primed panels without topcoat were also prepared by the NDCEE. Dry-film thickness measurements indicated that the primer was applied (average of four panels measured) at a dry-film thickness of 1.02/0.11 mil. Using this number, the film thickness of the topcoat can be estimated to be approximately 3 mil for series A, B, C, and E. The topcoat for series D was thinner, about 2.5 mil.

Performance test results were acceptable in most cases. ARL sent preliminary test results to TACOM in early June 1999. Because of problems with the spectrophotometer used to measure color and infrared (IR) reflectance, it was not possible at that point to calculate the color changes

Table 4. 4-in × 12-in Panel Dry-Film Thicknesses (mil)

Sequence No.	A	B	C	D	E
1	—[a]	—[a]	—[a]	—[a]	—[a]
2	3.95/0.15	3.82/0.22	3.68/0.39	3.50/0.30	4.37/0.26
3	4.01/0.31	3.91/0.15	3.70/0.37	3.42/0.44	4.41/0.22
4	4.01/0.22	3.88/0.22	3.68/0.23	3.48/0.33	4.45/0.32
5	3.96/0.12	3.78/0.14	3.77/0.27	3.44/0.26	4.29/0.31
6	3.94/0.15	3.84/0.26	3.52/0.37	3.53/0.38	4.43/0.28
7	3.97/0.11	3.78/0.15	3.69/0.34	3.46/0.30	4.37/0.24
8	3.87/0.27	3.65/0.21	3.58/0.32	3.46/0.36	4.24/0.28
9	3.78/0.25	3.68/0.18	4.17/0.39	3.30/0.34	4.13/0.24
10	3.68/0.23	4.09/0.15	4.20/0.33	3.24/0.26	4.15/0.30
11	3.71/0.28	3.94/0.20	4.20/0.28	3.37/0.30	4.10/0.26
12	4.01/0.28	4.00/0.26	4.18/0.38	3.44/0.37	4.08/0.23
13	4.10/0.16	4.08/0.30	4.07/0.39	3.36/0.29	4.02/0.30
14	4.04/0.23	3.85/0.18	4.28/0.34	3.44/0.50	4.09/0.26
15	4.13/0.24	3.93/0.26	4.07/0.37	3.43/0.33	4.05/0.24
16	3.96/0.19	4.02/0.25	4.20/0.25	3.44/0.32	3.87/0.34
17	4.04/0.22	3.85/0.26	4.07/0.31	3.36/0.15	4.11/0.29
18	4.16/0.24	4.20/0.24	3.95/0.33	3.39/0.35	4.04/0.30
19	4.22/0.35	4.00/0.33	4.05/0.31	3.33/0.36	4.11/0.38
20	4.15/0.37	4.01/0.30	4.38/0.35	3.48/0.29	4.02/0.31
21	4.28/0.30	3.97/0.32	4.00/0.31	3.37/0.26	4.06/0.37
22	4.04/0.13	4.06/0.22	4.18/0.35	3.55/0.31	4.15/0.29
23	4.22/0.27	3.93/0.16	4.03/0.37	3.34/0.37	4.09/0.31
24	4.09/0.31	3.89/0.35	3.97/0.28	3.27/0.35	4.04/0.29
25	4.13/0.22	4.15/0.25	4.02/0.34	3.34/0.27	4.56/0.31

[a]Sequence no. 1 panels were submitted for chemical-agent resistance testing prior to making the film thickness measurements.

due to accelerated weathering. In addition, the longer-term corrosion resistance testing was not yet complete. Since then, all testing has been completed, and the interim results can be updated. They are summarized in Table 6. Where appropriate, an elaboration on the test results is provided.

Table 5. 4-in × 6-in Panel Dry-Film Thicknesses (mil)

Sequence No.	A	B	C	D	E
1	1.31/0.19	1.63/0.13	1.67/0.04	0.89/0.14	1.28/0.14
2	1.37/0.14	1.61/0.15	1.53/0.16	0.94/0.11	1.28/0.17
3	1.28/0.13	1.68/0.1	1.60/0.10	0.96/0.09	1.37/0.19
4	1.24/0.12	1.66/0.08	1.44/0.21	0.98/0.10	1.26/0.15
5	1.32/0.07	1.58/0.13	1.43/0.23	0.92/0.06	1.36/0.09
6	—	1.59/0.12	—	—	—

Chemical-agent resistance was performed per paragraph 4.3.24 of MIL-C-53039 [4]. Results for two panels each series are complete, and the averages (in micrograms desorbed) for series A through E, respectively, are 40, 150, 10, 110, and 135. The allowable limit is 180.

Testing for color, gloss, IR reflectance, DS2 resistance, acid resistance, recoatability with MIL-C-46168 [3], recoatability with MIL-C-53039 [4], recoatability with MIL-P-64159 [5] (the proposed new specification for water-dispersible CARC), H_2O resistance, and HC resistance were done in accordance with the appropriate paragraphs in MIL-C-53039 [4]. This testing was essentially "pass/fail," and all results were acceptable. Testing for lubricating oil resistance and hydraulic fluid resistance was performed in accordance with the appropriate paragraphs in MIL-PRF-22750 [8]. Again, all results were acceptable.

Xenon-arc accelerated weathering was performed in accordance with American Society for Testing and Materials (ASTM) Standard G26 [9] in increments of 300 hr in a controlled irradiance Atlas Weatherometer. Color changes (an average of four panels exposed) after the first 300 hr, the specification requirement, were acceptable. The exposure has been continued for additional increments of 300 hr, and color changes at 600 hr and 900 hr are shown in Table 7.

QUV accelerated weathering was performed in accordance with ASTM G53 [10] for 500 hr in a Q Panel machine. Color changes (an average of four panels exposed) after 500 hr were acceptable. The exposure has been continued for additional increments of 500 hr, and color changes at 1,000 hr and 1,500 hr are shown in Table 7.

Table 6. UNICARB Test Result Summary

Test	Panel Series				
	A	B	C	D	E
Chemical-Agent Resistance	pass	fail	pass	pass	pass
Gloss	pass	pass	pass	pass	pass
Color	pass	pass	pass	pass	pass
IR	pass	pass	pass	pass	pass
1/4-in Mandrel Flexibility	pass	pass	pass	pass	pass
Spraying Properties	fail	fail	fail	pass	pass
DS2 Resistance	pass	pass	pass	pass	pass
Acid Resistance	pass	pass	pass	pass	pass
Recoatability	pass	pass	pass	pass	pass
Water Resistance	pass	pass	pass	pass	pass
Hydrocarbon Resistance	pass	pass	pass	pass	pass
Lube Oil Resistance	pass	pass	pass	pass	pass
Hydraulic Fluid Resistance	pass	pass	pass	pass	pass
Xenon Accelerated Weathering	pass	pass	pass	pass	pass
QUV Accelerated Weathering	pass	pass	pass	pass	pass
Outdoor Exposure	in progress	in progress	in progress	in progress	in progress
EMMAQUA	in progress	in progress	in progress	in progress	in progress
ASTM B117 [6] Salt Fog Exposure	—[a]	—[a]	—[a]	—[a]	—[a]
GM9540P Accelerated Corrosion	—[a]	—[a]	—[a]	—[a]	—[a]
Film Porosity	fail	fail	fail	pass	pass

[a]See Figures 1–4.

Table 7. Accelerated Weathering Color Differences (ΔENBS)

Method	Hours	Series A	Series B	Series C	Series D	Series E
Xenon	300	1.98	2.30	0.74	2.17	1.87
	600	6.98	5.96	4.41	7.66	5.67
	900	10.17	8.42	7.51	10.62	8.18
QUV	500	0.45	1.88	0.38	0.44	0.90
	1,000	4.30	5.85	3.08	2.67	3.91
	1,500	6.35	7.47	5.02	4.85	5.91

Although corrosion-resistance testing is generally not a topcoat test requirement, these tests were run to determine total system performance in both the standard ASTM B117 [6] salt fog exposure and in the GM 9540P [7] cyclic corrosion tests.

Twenty panels, four replicates for each of the five coating processes, were provided for ASTM B117 [6] testing. The lower half of two panels from each set were scribed with an X that covered the area. After the backs and edges were coated with beeswax, panels were exposed to 5% salt fog for 1,000 hr (~6 wk). Evaluations were performed using ASTM D1654 [11] procedure A for creep from scribe (direct measurements were done in millimeters, not the 1–10 scale provided, where 10 is the best) and ASTM D714 [12] for blistering. The creep from scribe, measured at the point of greatest creep, ranged from 3.75 mm for series C to 5.75 mm for series E, after 1,000 hr of exposure. This corresponds to ASTM D714 [12] ratings ranging from 5 to 4. The criterion for passing is a rating of 7 after 336 hr. Nearly all of the panels had blisters in the unscribed areas. Blisters in the scribed areas were ignored for this evaluation. Series A and series C each had one panel out of four that only had a few small blisters. The remaining panels exhibited varying degrees of poor performance. Series A, C, and E had bands in which concentrations of blisters occurred and other bands in which there were only a few or no blisters. This usually indicates that there is some variance in the coating application (thickness). Series B and D were each uniformly covered with blisters, indicating uniformly poor coating application or uniformly poor paint. Blistered areas for these two series is roughly the same, meaning that the performance is more similar than the ratings would lead one to believe. The topcoat specification allows there to be up to five blisters on a 4 in × 12 in after 336 hr of exposure to salt fog. When this requirement was written into the specification, preliminary testing indicated that blisters did not appear in greater numbers between 336 and 1,000 hr, just greater size. If ASTM D714 [12] is used to evaluate a panel, it has already failed the preceding requirement. As a basis for comparison, 260 panels were run for 2,000 hr of salt fog. One set of five panels was phosphated with MIL-P-53022 [2] and MIL-C-46168 [3] topcoat. Three of the five panels went through over 2,000 hr of salt fog with no blisters on any of the panels. The remaining two panels also did not have any blistering away from the scribe. Creep went from less than 1 mil after 336 hr, to about 3 mil after 1,000 hr, to 5 mil after 2,000 hr. This shows that, in spite of having twice the exposure time, the panels from the baseline study performed as well as or better than any of those provided for use in the UNICARB study. TACOM asked ARL to reexamine the blistered panels. Two panels were chosen at random and were immersed in deionized water for 24 hr to reactivate the blisters. Parallel cuts through the coating were made in two areas on each of the panels. Tape was applied and removed both with and across the cuts. Delamination occurred only on one small area of one of the cuts. A five-in-one tool was employed to scrape the coating from each of the panels. Based upon the preceding, the delamination is believed to occur at the metal/coating interface. The intercoat adhesion is very good, based upon the difficulties encountered while scraping. The blistering and creep evaluations are illustrated in Figures 1 and 2, respectively.

Twenty panels, four replicates for each of the five coating processes, were provided for GM 9540P [7] testing. The lower half of each panel was scribed with an X, while the upper half was untouched. After the backs and edges were coated with beeswax, panels were exposed to 100 cycles of GM 9540P [7]. During the course of the test, it was observed that the beeswax had melted and may have flowed on to the test panel surfaces, thus invalidating the test results. When this test is performed in future programs, beeswax will not be used. Evaluations were performed using ASTM D1654 [11] procedure A for creep from scribe (direct measurements were done in millimeters, not the 1–10 scale provided) and any corrosion anomalies were noted. The unscribed regions performed well in this test without the blisters that characterized the salt fog test. This may be because the GM 9540P [7] is a cyclic test that provides the coating an opportunity to dry. There were spots on each series of panels, except series E, where rust

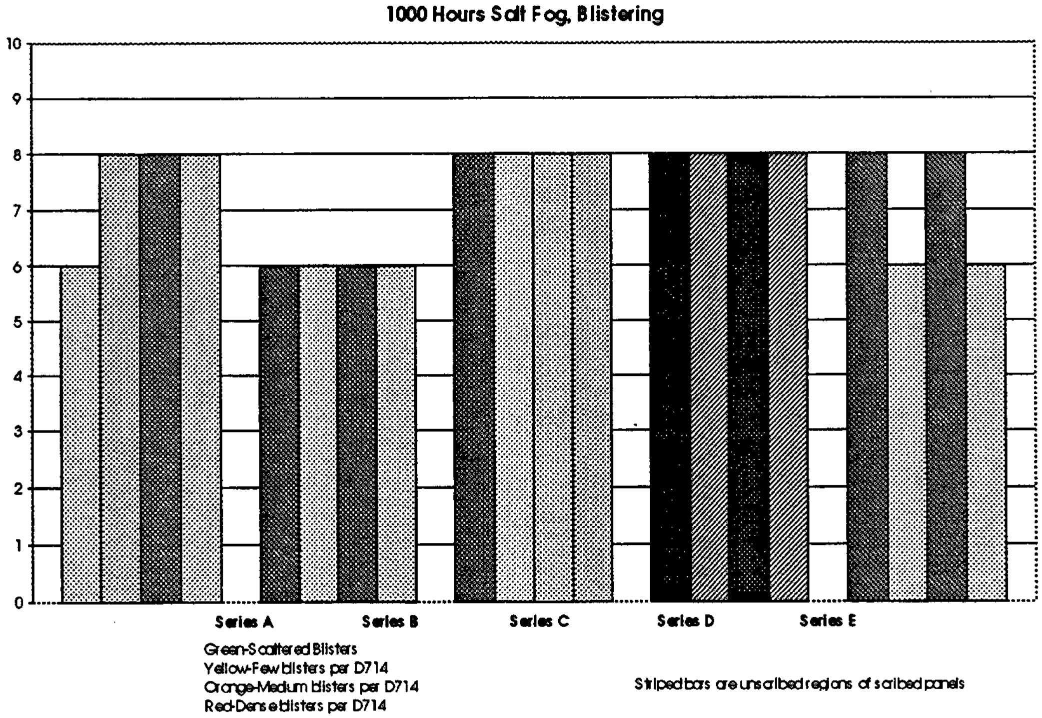


Figure 1. Salt Fog Blistering Results.

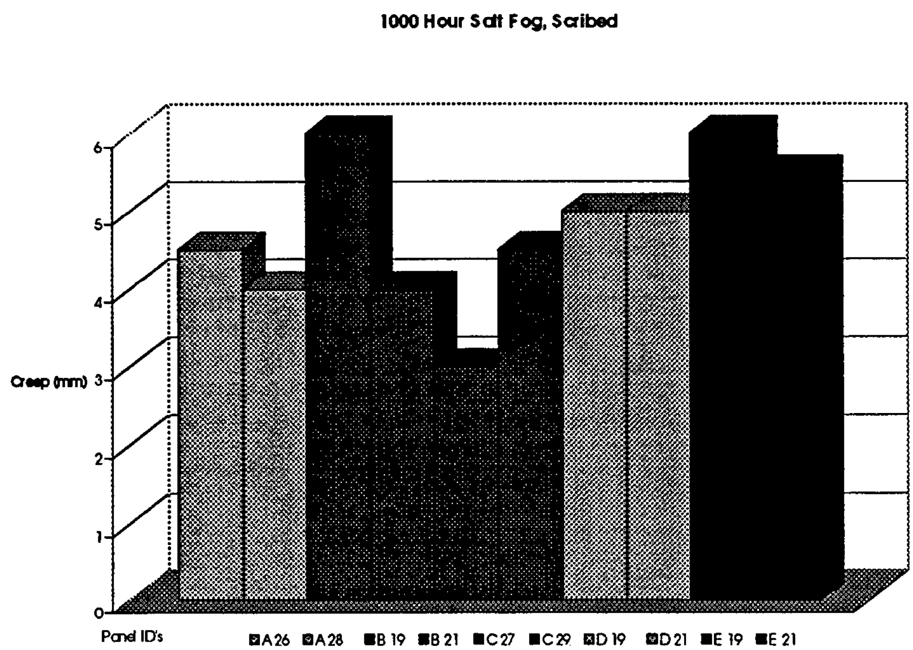


Figure 2. Salt Fog Creep Results.

bled through the coating. The most spots were seen on series B, followed by series C, A, and D. The problem is not particularly severe in that there were only four such spots on the most affected panel. However, this is probably indicative of a porous film that has borderline thickness thus allowing corrosive solution to the substrate in a short period of time. These features, once present, did not grow in size. The performance in the scribed regions exhibited distinct trends. At the halfway point of the test, series C had the greatest creep from scribe, with an average value of 3.35 mil. Series A and B followed with values of 2.5 mil, and series D and E each had averaged values of under 2 mil. The trend continued at 75 cycles, with series C turning in the worst performance, followed by series A and B. Series D and E continued to do best. At the conclusion of the test, series C had an averaged creep from scribe of 7.1 mil, followed by series A at 6.1 mil. Series B, D, and E performed comparably, with averaged creep of under 5 mil. The panels in this study had less creep from scribe than comparable panels in the water reducible polyurethane matrix. Averaged creep was greater than 10 mm. However, these panels did not have any of the rust spots on their relatively small, unscribed areas. This performance is consistent with an even nonporous primer/topcoat layer, where all damage occurring at a manufactured coating defect at the metal-pretreatment interface. The choice of beeswax as a back and edge protector for GM 9540 will be avoided in the future, since the temperature reached in the high temperature dry off cycle causes the wax to soften or melt. The lower melting fractions may have helped protect the test surfaces of the panels by filling in porosity or by flowing into the scribe. Averaged performance and creep evaluations are illustrated in Figures 3, 4, and 5, respectively.

As noted in section 1, all of the panels prepared by supercritical CO_2 reduction had poor film properties, indicated by excessive voids and pinholes. Appearance problems included nonuniform color and gloss, and what appeared to be areas showing a "dry spray" appearance. In addition, closer inspection of these panels indicated that film porosity could be a problem. Examination under minimal magnification revealed apparent holes in these films that were not visible in films that had been applied conventionally, either with or without solvent reduction. These defects alone were sufficient to fail the panels on spraying properties. However, in order

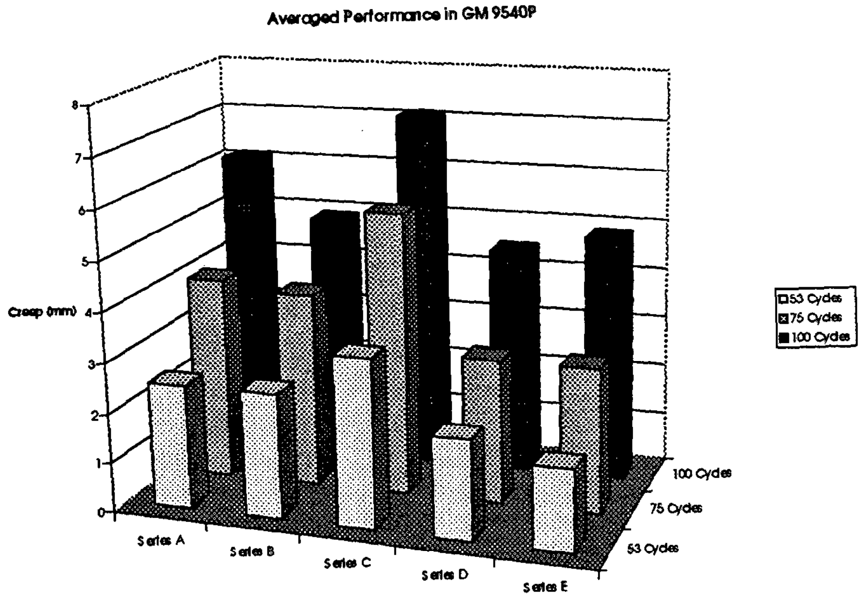


Figure 3. GM 9540P [7] Performance Results.

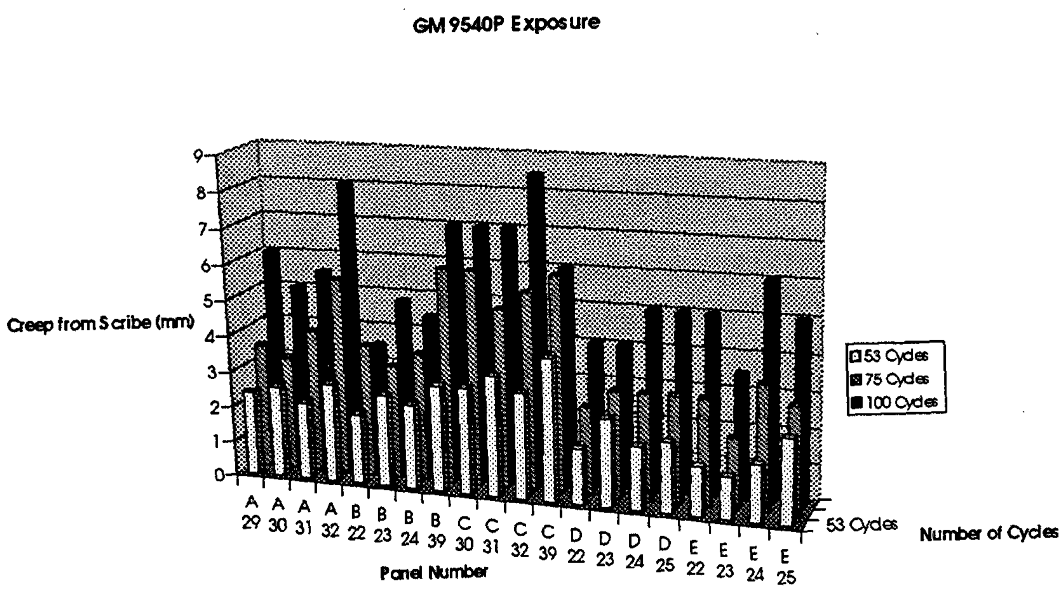
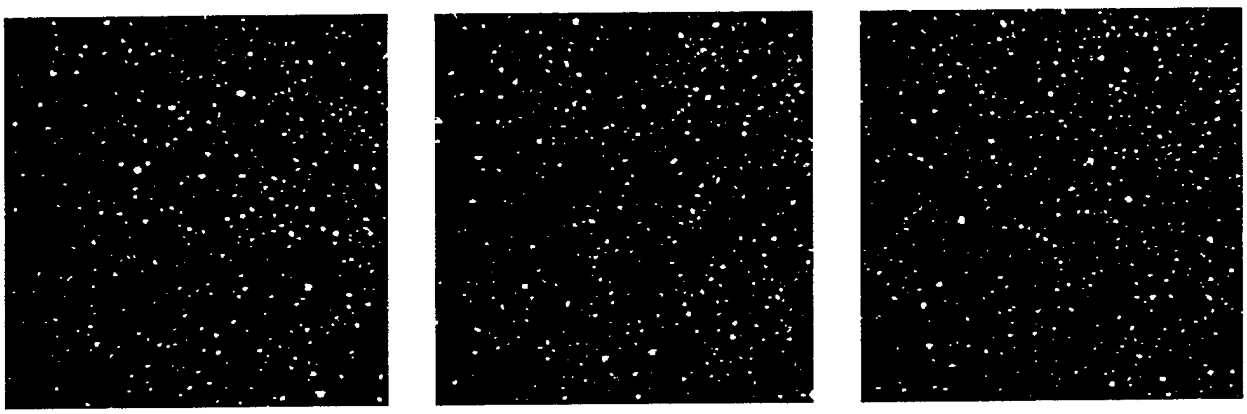


Figure 4. GM 9540P [7] Creep Evaluation Results.

A Panels



B Panels

C Panels

Figure 5. Black and White Images.

D Panels

E Panels

Figure 5. Black and White Images (continued).

to provide a less subjective means to compare all of the dried paint films, color photographs were electronically manipulated into black and white renderings in which the paint film was black and the holes were white. The following procedure was used to develop these images.

(1) All shots were done at 320× magnification, using a Wild Stereo Microscope, and lighted using an 8-in ring light.

(2) The color camera was in Y/S-video mode.

(3) The micrographs were histographically adjusted to include the full tails of each histograph.

(4) The micrograph was duplicated, and then adjusted to the peak of the histograph, converted to black and white and readjusted to that histographic peak.

(5) The image was then inverted, followed by threshholding the image at 200 (out of a 256-level grey scale), where 0 = black and 255 = white.

(6) All image manipulations were performed using PhotoShop 5.02.

Figure 5 illustrates the differences between the five panel sets.

4. Discussion

TACOM and ARL's mutually developed test matrix helps bring to light the poor film application ability of supercritical CO_2 with pigmented coatings and the subsequent detrimental effects the porous film may impart upon the coating system in a corrosive environment. While many critical tests did pass, it is essential that all requirements be met to be an approved coating.

5. Conclusion

Detailed conversations between TACOM and ARL team members have resulted in requesting Union Carbide and Hentzen to rework both the formulation and application procedures to produce a void-free CARC film and to implement and provide an initial transfer efficiency baseline with the reformulated coating. This two-step approach would assist all interested parties as to the practicality and economical feasibility of implementing the supercritical CO_2 process. Until this is accomplished and all performance requirements are met, the application CARC using the UNICARB (supercritical CO_2 reduction) will continue to remain an unacceptable process and unapproved coating for the Army.

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Jeffrey Duncan, Donovan Harris, and Christopher Miller

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U.S. Army Research Laboratory
ATTN: AMSRL-WM-MA
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At the request of the U.S. Army Tank-automotive and Armaments Command (TACOM), the Coating Technologies Team of the U.S. Army Research Laboratory (ARL), Weapons and Materials Research Directorate (WMRD), evaluated a chemical-agent resistant coating (CARC) reformulated to be compatible with the supercritical CO_2 application process. Test panels were prepared by the Concurrent Technologies Corporation (CTC) and submitted to ARL's Experimental Products Program (EPP) for approval. Thus far, four different and separate formulations have been submitted, and all have failed, including the fourth and most recent submission prepared at the CTC on 12 November 1999. In each case, excessive voids and pinholes riddled the coating film applied with supercritical CO_2. To date, the use of supercritical CO_2 has not been successful in demonstrating its ability to apply CARCs free of voids or pinholes. This report summarizes the test program and the results through 24 November 1999.

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