E. Aluminum Automotive Closure-Panel Corrosion Test Program (AMD 309[i])

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Objective

- Develop a standardized cosmetic corrosion test for finished aluminum (Al) automotive body panels that provides a good correlation with in-service testing and field performance.

Approach

- Conduct laboratory testing, outdoor exposures, test-track exposures and in-service testing.
- Evaluate test data to determine which accelerated tests correlate with in-service testing.
- Conduct iterative laboratory testing to improve correlation between lab tests and on-vehicle exposures.

Accomplishments

- Initial laboratory tests, test-track exposures and outdoor exposures completed
- On-vehicle tests have been exposed for three years out of five planned.
- Corrosion product analyses conducted for some laboratory tests and for two-year exposures on-vehicles.
- Round-robin testing conducted for the three lab tests down-selected from initial lab tests.
- Conducted an initial evaluation of three existing ASTM tests with sulfur in the exposure.

Future Direction

- Modifications of the more promising accelerated laboratory tests are now being considered in an effort to find a lab test with improved correlation to on-vehicle exposure results in terms of the extent and morphology of the corrosion as well as the composition of the corrosion products. On-vehicle exposures will continue until the panels have been exposed on vehicles for five years.

Introduction

Although Al closure panels have been used on numerous vehicles for several years, the degree of confidence in predicting service performance has not been high due to the lack of an accelerated corrosion test that mimics field performance. Automotive manufacturers and their suppliers often rely on accelerated corrosion tests that were developed for evaluating steel, but these tests are not always consistent with in-service Al closure-panel performance.

In order to address the need for an accelerated Al corrosion test, a group comprised of representatives from the US automotive manufacturers, Al suppliers, coating suppliers, and other associated suppliers was formed in 2000. The goal of this group has been to identify and implement a standardized, accelerated corrosion test for cosmetic corrosion of Al that exhibits the same appearance, severity, and corrosion products that are exhibited on in-service Al components.

Experimental

Materials

In 2001, the first step in the development of a new cosmetic corrosion test occurred with the establishment of a reservoir of painted panels. These panels would then be used in the subsequent evaluation of all test methods. As listed in Table 1, the substrate materials, metal finish and paint processing variables were selected to give a range of cosmetic corrosion performance. Several Al alloys used in the Unites States and in Europe, both current and historical, were included. Electro-zinc-coated steel and uncoated cold-rolled steel were included as reference materials. Two Al alloys were processed to simulate metal finishing in an automotive assembly plant body shop. Two sizes of panels, 2" x 4" and 4" x 6", of each of the materials were painted with a typical automotive paint system. This paint system included zinc-phosphate pretreatment, medium-build cathodic electrophoretic priming (E-coat), and spray painting with a primer surfacer and white basecoat/clear topcoat system for a total paint film thickness of approximately 100μm. One set of 6111 panels (Panel Code B) was processed through the phosphate pre-treatment with lower fluoride concentration (comparable to the fluoride level used for steel-only vehicles which results in lower phosphate coating weight). Also, since qualification testing is often done on panels that are processed only through the electrophoretic primer (E-coat) step, another set of 6111 panels (Panel Code C) was processed only through the E-coat step (i.e., standard fluoride for Al but no basecoat or clear-coat applied). Panel code C was evaluated in accelerated tests only (no on-vehicle or proving ground exposures).

Table 1. Materials.

Panel Code	Alloy Substrate	Metal Finish	Paint System
A or 1	AA6111-T4PD	Mill	Standard
B or 2	AA6111-T4PD	Mill	Low F-
C or 3	AA6111-T4PD	Mill	Ecoat Only
D or 4	AA6111-T4PD	Sanded	Standard
E or 5	AA6016-T4	Mill	Standard
F or 6	AA6022-T43	Mill	Standard
G or 7	AA2036-T4	Sanded	Standard
H or 8	EG 60 Steel	Mill	Standard
I or 9	Cold Rolled Steel	Mill	Standard

Panels were prepared as-needed for testing with two parallel scribes penetrating through the coatings to the substrate. The panels were provided to the testing laboratories as fully-prepared painted and scribed panels. Triplicate sets of the painted and scribed samples have been exposed in a variety of environments, including laboratory, static outdoor exposure, proving-ground, and on-vehicle tests.

Evaluation Method

For this study, an optical imaging system developed by Atlas Material Testing Technology LLC was employed to quantitatively interpret the degree of cosmetic corrosion. The imaging system employs controlled illumination conditions, high-resolution digital-image capture and advanced algorithm-based image- and data- analysis methodologies. [1].

Four geometrical attributes of the cosmetic corrosion were measured: area of corrosion, maximum length, minimum length and average length. The area of corrosion was found to be the most representative and comprehensive measurement. Because the size of the panels and therefore the scribe lengths for the lab tests were different from the other tests, the corrosion area was normalized to the length of the scribe (i.e., area per length). The normalized

corrosion areas for triplicate panels (2 scribes per panel) were then averaged and the results are reported as "normalized average area".

Laboratory Tests

In the initial round of testing, each of the tests listed in Table 2 was conducted at two laboratories as a limited check on the lab-to-lab reproducibility of the initial results. After comparing the initial round of test results to preliminary on-vehicle exposure results, three of these tests (Ford Arizona Proving Grounds Exposure (APGE) [2], ASTM G85 - Annex 2 [3], and HCl Dip [4]) were selected for further evaluation.

Round-robin evaluations of these test methods have been conducted. The duration of the tests was varied in an attempt to further improve the correlation of these accelerated tests with the results from the on-vehicle exposures. Each test was conducted at four to five laboratories to more thoroughly evaluate the repeatability and reproducibility of the test methods. Three additional tests were added in the second round of testing to include exposure to sulfur. They were ASTM G85 Annex 4 [5] ASTM G85 Annex 5 [6] and ASTM G87 [7]. A listing of the accelerated laboratory tests that were run in the second round of testing is given in Table 3.

Table 2. Laboratory Test Evaluated in Initial Round of Testing.

Test Procedure	Exposure (s)
SAE J2334	40, 60 & 80 cycles
GM 9540B	40, 80 cycles
Ford APGE	35 & 70 cycles
ASTM D2803 (50,80 & 100% RH)	6 weeks
ASTM G85 Annex 2	3 weeks
VDA 621-415	10 cycles / 70 days
ASTM B117	500 & 1000 hours
CCT IV	70 cycles
HCl Dip	8 weeks
KWT	6 weeks

Table 3. Laboratory Test Evaluated in Second Round of Testing.

Test Procedure	Exposure (s)
Ford APGE (Manual & Automated Humidity Cycle)	70 cycles
ASTM G85 Annex 2	1 & 2 weeks
HCl Dip	3 & 6 weeks
ASTM G87	20 cycles
ASTM G85 Annex 4	500 hours
ASTM G85 Annex 5	500 hours

On-Vehicle (In-Service) Exposures

It is critical when developing a laboratory-based test that test-to-field correlation be performed. In an effort to capture real-world data in developing this test, it is necessary to expose these panels to severely corrosive environments that represent "worst case" real-world service environments. Suitable environments exist in the northeastern United States, southeastern coastal areas of the United States, and southeastern Canada. The five sites selected for this study were: 1) Detroit, Michigan; 2) Florida; 3) St. Johns, Newfoundland; 4) Montreal, Quebec; and 5) an Ohio-to-New York truck route.

Two sets of 2" x 4" test panels are exposed on each vehicle (two vehicles per site). Each set of 24 panels (three replicate samples of each of the eight material variations) are attached to a mounting panel (16" x 12") using double-backed tape prior to mounting on the vehicle. At the Detroit, St. Johns, and Montreal sites, one set is mounted on the hood of each vehicle (horizontal orientation) and one set on the right front door of each vehicle (vertical orientation). At the Florida and Ohio-New York sites, the panels are mounted beneath the trailer frame behind the front wheels (vertical orientation only). Each panel contains 2 diagonal scribe lines which are 2" long and 1" apart. The panels will be exposed for a total of five years of in-service exposure. Intermediate evaluations will be conducted when possible.

OEM Test-Track Exposure

Completed OEM test-track results were published previously [8] and are not repeated in this report.

Outdoor Exposure

Completed outdoor exposure test results were published previously [8] and are not repeated in this report.

Corrosion-Product Analysis

The corrosion products from selected on-vehicle exposure panels have been analyzed using a variety of electron-optical techniques. Select panels were removed from the on-vehicle exposures at three locations (St. Johns, Montreal, and Detroit) after two years of exposure for compositional analyses of the corrosion products.

Top-down analysis of the corrosion products was carried out after stripping the surrounding paint layer in 1-methyl-2-pyrrolidone solvent. Water was not used during the stripping procedure so as to preserve any chloride/sodium species present within the corrosion product. The panels were examined using a Leo 440 scanning electron microscope (SEM) equipped with a Quartz XOne energy dispersive X-ray (EDX) analysis system. SEM/EDX technique was chosen for this examination because the amount of corrosion product on Al closure panels is very small, making more traditional analytical procedures impractical. In addition, the use of SEM/EDX allows not only for a measurement of the chemical species present, but also for an analysis of the distribution of these species in and around the corroded area on the panel.

Results and Discussion

Lab-Test and On-Vehicle Results

At this point in the program a limited amount of service-relevant results from the on-vehicle exposures is available. Although there is significant variability both from panel-to-replicate-panel and from vehicle-to-vehicle for a particular exposure site, the most consistent result is that panels D, G, and I exhibited more corrosion than the other substrates as is illustrated by the images in Figure 1 of a set of panels from the Detroit on-vehicle exposures after being exposed for approximately eighteen months.

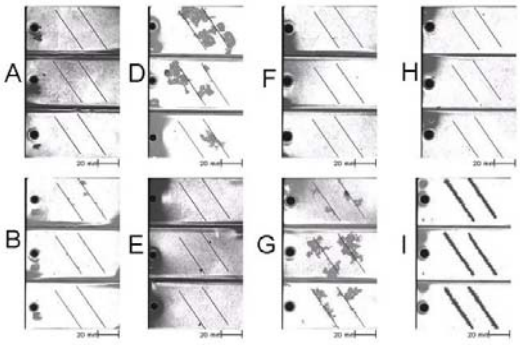

Figure 1. Detroit On-Vehicle Test with 1.5 Years Exposure.

From the preliminary on-vehicle results it is apparent that the panels with metal finishing (D & G) generally have more corrosion than the other Al substrates and that the cold-rolled steel (I) has more corrosion than the electro-galvanized steel (H). The normalized average area from on-vehicle service relevant exposures in Detroit and Montreal are plotted in Figure 2. Based on these preliminary on-vehicle results, accelerated tests that also show a significant difference between the substrates with metal finishing (i.e., sanding) and those without metal finishing would appear to correlate better with the preliminary observations from on-vehicle testing. Many of the accelerated lab tests in the initial round of tests did not show a significant difference between the substrates with and without metal finishing. Of the initial set of laboratory test methods evaluated in the program, only the APGE (one lab only), ASTM G85-A2 test and HCl dip tests appeared to show a significant difference in corrosion performance between the substrates with metal finishing (i.e., sanding) and those without metal finishing.

Because these three accelerated tests had shown signs of possible correlation with on-vehicle exposures, they were selected for further evaluation. In an attempt to improve the correlation of the type and extent of corrosion, reduced durations of the HCl Dip (three- and six-week) and ASTM G85-A2 (one- and two-week) exposures were evaluated. The tests were also conducted as round-robins with a minimum of four laboratories to further evaluate the reproducibility of the tests. Also, in order to determine if the divergent results from the APGE

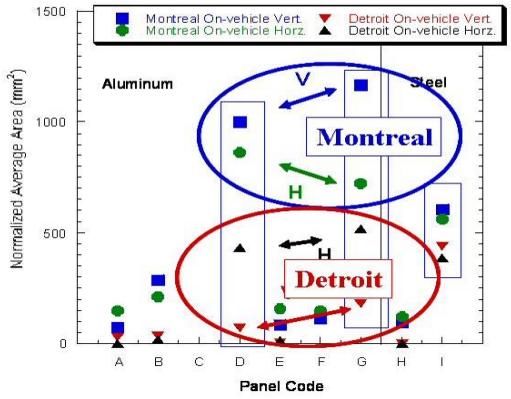

Figure 2. Normalized average area: Detroit and Montreal with two years exposure.

tests were associated with means of cycling (i.e., automated vs. manual), the 70-cycle APGE test was repeated at six laboratories, three with manual cycling and three with automated cycling.

In Figures 3 and 4 the results from the ASTM G85-A2 round-robin are shown with the results from the worst-case on-vehicle exposure results to date, Montreal vehicle #2.

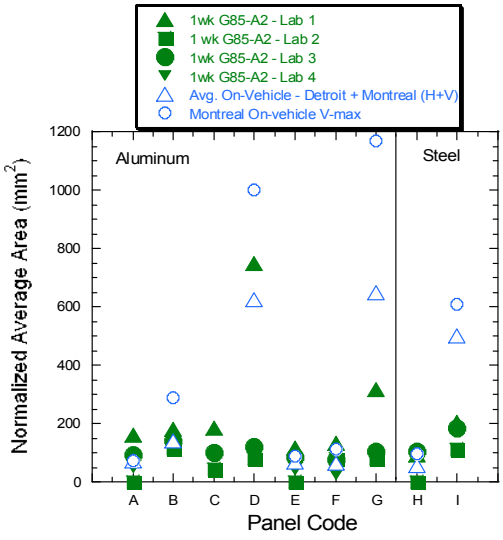

Figure 3. ASTM G85-A2 round-robin tests – 1-week with results from the worst-case on-vehicle (Montreal).

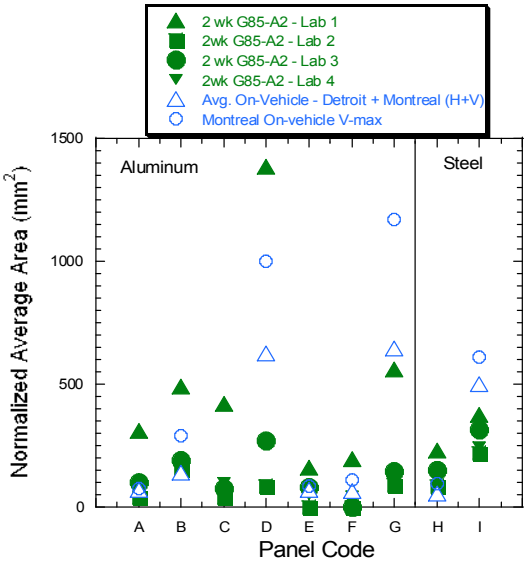

Figure 4. ASTM G85-A2 round-robin tests – 2-weeks with results from the worst-case on-vehicle (Montreal).

The relative ranking of the panels in the two-year Montreal on-vehicle were slightly different for the panels exposed vertically on the doors and those mounted horizontally on the hoods:

- Vertical = G>D>>I>B>rest
- Horizontal = D>G>I>>B>rest

The ASTM G85-A2 results for Lab #1 were similar to the previous results in the initial round of testing but significantly different from other labs in the round-robin. The relative ranking of the panels in ASTM G85-A2 is as follows: Lab #1 is reasonably similar to that from the Montreal vehicle:

- 1 week – Lab #1 = D>G>rest
  (Other labs no differentiation)
- 2 weeks – Lab #1 = D>>G, B, C, A>rest
  (Other labs little to no differentiation)

For Lab #1, the relative ranking of the Al substrates is reasonably similar to that from the Montreal vehicle as was noted in the initial tests. The lack of differentiation for the other three labs is an indication that the actual test conditions were substantially different. After these results came to light, we reviewed the actual conditions at each lab. A detailed scrutiny of the test specification revealed parts of the test description could be readily misinterpreted and that the actual chamber

conditions are very specific and may not be able to be run on all standard corrosion equipment.

Figures 5 and 6 show the results of the HCl dip test round-robin with the results of Montreal in-service vehicle #2. The HCl dip test was run for two exposure time periods (3 weeks and 6 weeks) at four laboratories. Overall, the 3-week exposure does not show as much corrosion as seen in the field. Therefore, it is unlikely that the 3-week test is long enough to characterize in-service corrosion performance.

For the 6-week HCl dip test, the corrosion performance on substrates D and I is much closer to that seen in the on-vehicle exposures. However, two of the laboratories (lab #2 and lab #4) significantly over-predict the corrosion on substrates A, B, and H compared to the 2-year in-service performance. This over-prediction occurs only at two of the four laboratories.

The other two laboratories (lab #1 and lab #3) follow the corrosion trends seen in the on-vehicle exposures, with the only exception being for substrate G. Further investigation is needed to understand the test conditions at the laboratories running the HCl dip test to explain the differences in the results after six weeks of testing.

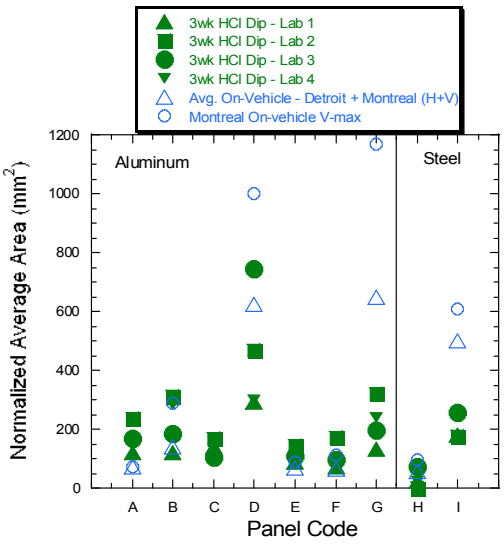

Figure 5. HCl dip round-robin tests – Three-weeks with results from the worst-case on-vehicle (Montreal).

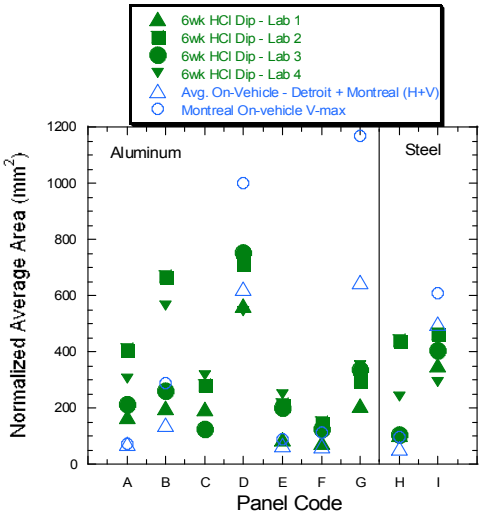

Figure 6. HCl dip round-robin tests – Six-weeks with results from the worst-case on-vehicle (Montreal).

For the second round of APGE (70 cycles), tests were conducted at a total of six labs, three with manual cycling and three with automated cycling. The results from four of the six labs are available at this time and are shown in Figure 7. The results from one test lab at which the cycling was conducted manually appears to differentiate between substrates D, G, and I and the remaining substrates thereby showing reasonable correlation to the on-vehicle exposures but the other three labs do not show this differentiation.

In the first round of tests, only the lab that had run the test with automated cycling seemed to differentiate the substrate groups. At that time, the lack of lab-to-lab reproducibility for the APGE test was suspected to be related to differences between manual and automatic cycling, but these more recent results contradict the earlier results and indicate that there are other, less obvious, sources for the lab-to-lab variability in the results. Further investigations will be carried out to understand the test conditions at the laboratories running the APGE test to explain the differences in the results.

The limited results from the two sulfur bearing tests are shown in Figures 8 and 9. To date, these tests have only been run at one lab each. The results for the salt/SO_2 fog test (ASTM G85-A4) show significant corrosion on most of the panels.

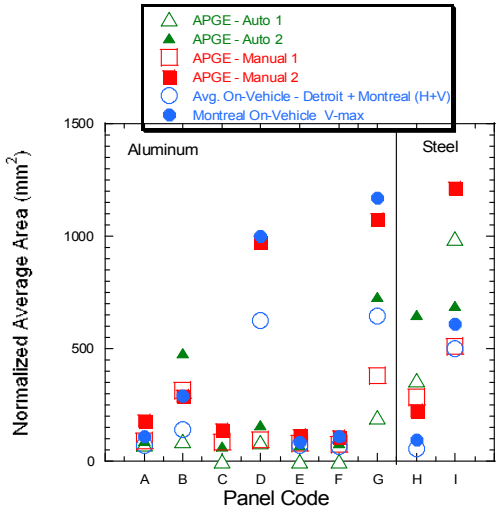

Figure 7. Ford APGE round-robin tests – 70 Cycles with results from the worst-case on-vehicle (Montreal).

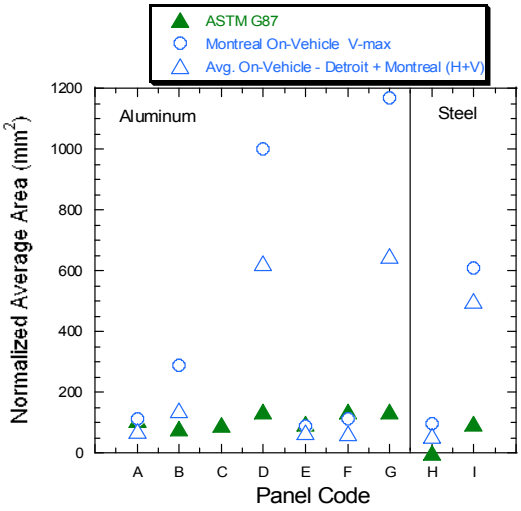

Figure 9. ASTM G87 round-robin tests – 500 hours with results from the worst-case on-vehicle (Montreal).

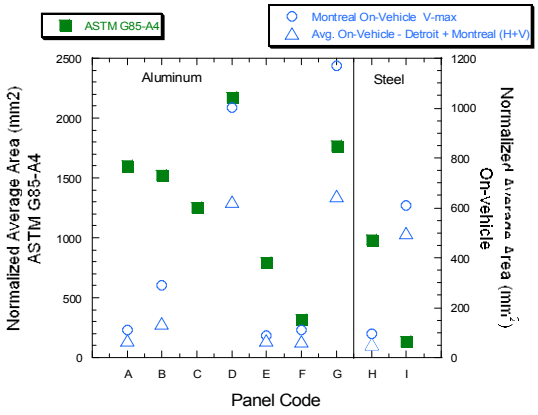

Figure 8. ASTM G85-A4 round-robin tests – 500 hours with results from the worst- case on-vehicle (Montreal).

Although the D and G substrates have the most corrosion, this test method as conducted does not appropriately differentiate D and G from the other substrates (A, B, E) which have far less corrosion in the on-vehicle exposures than was observed in the ASTM G85-A4 test. This test method also appears to be more severe for galvanized steel than for cold-rolled steel which is opposite of the on-vehicle exposures as illustrated in Figure 8.

The lone set of results for the moist SO_2 test (ASTMG87) illustrated in Figure 9 shows very little corrosion on any of the substrates and does not appear to have differentiated the substrate groups in the manner observed in the on-vehicle tests.

Corrosion-Product Analysis Results

Table 4 presents a description of Al panels examined in this study. These panels came from three different in-service exposures (St. Johns, Detroit and Montreal). SEM/EDX was performed at several areas on each panel. Only selected, but representative, results from this investigation are presented here. EDX spot analysis was performed at several locations to determine the variations in the corrosion-product composition from location to location.

Table 5 presents the EDX spot analysis results from various locations within the corroded region for representative panels from the St. John's exposure. Sulfur was detected on all three Al panels, whereas chlorides were detected on panels A and G only. Moreover, the tip of the filiform filament (spots SA1, SG2 etc.) exhibited much greater concentrations of chlorides species. In general, the average sulfur values, calculated from ten to fifteen spot analyses on each panel, were greater than the average chloride values for the Al panels.

Table 6 presents the results from panels A and D exposed in Montreal. The results from the spot analysis indicate that chloride-rich areas were detected in the corroded region as well as in the scribed region. In contrast, sulfur-rich areas were not observed on panel A and only at the scribed region on panel D.

Table 4. Description of Panels Selected for Corrosion Product Analysis.

Location	Panel Code	Panel Description
St. John's, Newfoundland	A	Vehicle 3 rep 1H top hood 3
	D	Vehicle 3 rep 3H bottom hood 2
	G	Rep 1 H top hood 3
	I	Ford-061 Vertical
Detroit, Michigan	A	Ford-061 Horizontal
	B	Ford-061 Horizontal
	D	Ford-061 Horizontal
	D	Ford-061 Vertical
	I	Ford-061 Horizontal
	I	Ford-061 Vertical
Montreal, Quebec	A	Field Motor Vehicle 2
	D	Vehicle 3 rep 1H top hood 3

Table 5. Composition of Corrosion Products on Panels from St. John's Exposure (weight %).

Spot	O	Al	P	S	Cl	Ca	Zn
SA1	56.3	33.2	2.7	3.2			1.3
SA2	61.5	31.5	0.7	2.3	2.6		0.9
SA3	9.7	8.0	2.7	0.4	0.1		6.1
SA4	58.2	30.4	1.0	0.8	0.5		7.6
SA5	25.6	69.7	1.2	1.0	0.1	0.3	1.6
SD1	60.4	31.0	2.2	3.0		0.2	2.7
SD2	60.1	29.8	3.1	2.6		0.2	4.2
SD3	46.2	37.8		1.2			
SA9	40.6	31.3	7.9				15.5
SG1	46.5	27.0	6.8		2.4		3.2
SG2	58.6	29.1	3.2	0.1	0.4		1.3
SG3	47.1	20.8	8.7		2.3		3.6
SI1	7.1						91.4
SI2	31.6		0.1	0.2		0.3	52.2
SI3	26.4	0.7			0.3	4.2	6.0

EDX spot analyses were performed at ten different locations on panel A and sixteen different locations on panel D, though the data from only three to four locations are presented in Table 6. The trends from these extensive EDX spot analyses indicated that the Montreal exposures exhibited more chloride-rich regions than the sulfur-rich areas. This observation is in contrary to the data from St. John's panels.

Table 6. Composition of Corrosion Products on Panels from Montreal Exposure (weight %).

Spot	O	Na	Al	P	Cl	Ca	Zn
MA1	54.3	4.6	34.6	1.2	1.5	3.1	0.1
MA2	49.4	1.6	30.2	10.1	2.5		
MA3	48.3	3.1	30.1	4.9	1.0	4.1	6.3
MA4	34.0		13.1	11.4			35.2
MD1	54.4	2.9	29.9	5.7		7.0	
MD2	63.4	1.0	33.3	1.1		0.7	
MD3	46.8	1.6	26.5	5.8		2.5	13.7

Table 7. Composition of Corrosion Products on Panels from Detroit Exposure (weight %).

Spot	O	Al	P	S	Cl	Zn	Fe
DA1	59.6	28.5	1.7	3.8	3.5	2.4	
DA2	54.9	33.1	1.9	4.6	0.2	2.8	
DB1	58.4	39.1	0.3	1.1	1.0		
DB2	55.4	40.9	0.2	1.7	1.5		
DDH1	57.3	32.0	3.9	1.5		4.3	
DDH2	6	31.9	2.4	1.9	0.2	3.0	
DDV1	61.8	27.7	1.0	4.0	0.9		
DDV2	35.6	61.6	1.2				
DIH1	24.7				3.5		71.3
DIH2	34.4				0.4		64.6
DIV1	40.4			0.4	2.3		55.3
DIV2	43.5				0.5		56.0

Finally, EDX spot analysis data from the Detroit exposures are summarized in Table 7. The data from panel A indicate that the corroded region was enriched with sulfur. Chloride concentrations were also detected at the tip of the filiform filament. EDX spot analysis data (Table 7) also shows that all the spots exhibited sulfur or chloride or both. Sulfur and chloride enrichment was also observed on panel B. EDX elemental distribution maps for horizontal and vertical D panels indicate that the horizontal panel exhibited sulfur within the corrosion product, while both sulfur and chlorides were detected at the corroded region of the vertical panels. However, the EDX spot analysis data shown in Table 7 indicate that small amounts of chlorides could be detected within the corroded region of the horizontal panel.

Conclusions

Correlation of the accelerated corrosion tests to on-vehicle corrosion performance is the primary goal of this test development effort. From the preliminary on-vehicle results it is apparent that the panels with metal finishing (D & G) generally exhibit more corrosion than the other Al substrates In the first round of accelerated corrosion testing, only three test methods (APGE, HCl Dip, and ASTM G85-A2) were able to distinguish substrates D and G from the other Al test samples. However, the corrosion morphology on those substrates did not appear to be the same as that found on the on-vehicle panels.

In the corrosion product analyses of the on-vehicle panels, both sulfur and chloride were detected to varying degrees. Overall, the Al panels appeared to exhibit greater amounts of sulfur species within the corrosion products than the steel panels that were exposed to the same environment. This would seem to indicate that the Al panels were more sensitive to the presence of sulfur and that sulfur plays a more significant role in the corrosion of Al than in the corrosion of steel. As the first round of accelerated testing did not include a test that incorporated an exposure to sulfur, a second round of testing was run that included tests with sulfur species in the exposure (ASTM G87, ASTM G85-A4, and ASTM G85-A5).

The results of ASTM G87 showed very little corrosion on the Al panels. As this test provided exposure to moist SO_2 and no exposure to chloride, it would seem to indicate that either corrosion on Al does not occur with exposure to SO_2 alone or that another sulfur species may be responsible for corrosion on Al.

ASTM G85-A4 is a salt/SO_2 fog test. Al panels that underwent ASTM G85-A4 exhibited significant corrosion on many of the panels. Although the test looks promising for the prediction of corrosion on substrates D & G, it significantly over-predicts the corrosion on substrates A and B when compared to the on-vehicle panel exposures. Further evaluation of this test is needed to determine if the balance of salt and SO_2 or other test parameters can be optimized to provide a better correlation to field performance for all of the Al substrates being tested.

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Presentations/Publications/Patents

SAE 2002B-203
SAE 2003-01-1235
SAE 2005-01-0542
SAE 2006 Oral Only

[i] Denotes project 309 of the Automotive Metals Division (AMD) of the United States Automotive Materials Partnership (USAMP), one of the formal consortia of the United States Council for Automotive Research (USCAR), set up by the "Big Three" traditionally USA-based automakers to conduct joint pre-competitive research and development.