2012

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An Evaluation into the Cause of Corrosive Failure in Autophoretic Coated Material

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ABSTRACT

This paper documents a study of performance failures in Autophoretic® (A-coat or AP) coatings. As AP is a proprietary process and coating material, limited research has been published regarding the field performance of this coating technique. The specific failure under analysis in this study was corrosion. The study was performed at the request of a manufacturer that was experiencing pre-mature failures in the field on product coated using this process and material. Prepared samples were evaluated using several analysis techniques including, BET Gas Absorption Testing, Optical Microcroscopy, FE-SEM, and Corrosion-Rate Analysis. Samples were subjected to the Society of Automotive Engineers (SAE) – Surface Vehicle Standard, J2334 Cosmetic Cyclical Corrosion Lab Test to accelerate the corrosion process, simulating long-term field conditions. Micro-cracks and pores were identified in the final finished surface that proved to be the point of origin of extensive corrosion that was the result of creep under the surface of the finish and ultimate delimitation.

KEYWORDS

Autophoretics (A-coat or AP), Powder Paint, Corrosion, Green Manufacturing

INTRODUCTION

Autophoretics®; also known as Autodeposition, A-coat or AP, is a waterborne process which depends on chemical reactions between a ferrous component to be coated and the coating solution. Iron reacts with a mildly acidic latex emulsion polymer, as well as other undisclosed ingredients. These ingredients vary depending on manufacture, usage, and desired properties. The mild acidity releases a small amount of ions from the ferrous material. These ions interact with the latex causing a thin deposition layer to form. The chemical activators diffuse rapidly into a film and etch the surface simultaneously. Unlike an electrostatic coating, there is no external electric charge and the pigment is in solution. This process is only possible with ferrous materials. Once the coating is applied it is heat cured to solidify the coating onto the base substrate. [1, 2, 11]

Due to the proprietary AP process and material, there is limited research that has been published regarding this coating method. Of the limited research, a few notable studies must be mentioned include a comparative study between autophoretics and cataphoretic coatings [13]. The authors found the AP coating to have comparable corrosion resistance to cataphoretic coatings in the study; of which both were found to have acceptable corrosion resistance in automotive applications. Another study previously evaluated the effects of bath temperature on autphoretic deposition. It was found that the a reduction of coating film quality when bath temperature increased above 20°C [14]. Other than these few research studies, there has been no other studies investigating or identifying potential causes for corrosion in AP coatings to the authors knowledge. Based on this information, it was merited to conduct an investigation of potential causes for corrosive failures in AP coatings.

The AP coating film thickness is time and temperature dependent. The coating process will continue as long as there is a metal/solution interface and ferrous ions are produced. As a coating thickness increases the interface diminishes. Therefore the coating rate is initially rapid then decreases as the coating thickness increases. Typically, coating thicknesses are between 0.6 and 1.0 mil but can reach as high as 1.9mil under controlled conditions. [1-3]

One of the primary advantages of A-coat is that Volatile Organic Compounds (VOC) emissions are extremely low. Depending on the resin being used it is possible to have zero VOC emissions. [3, 5] Another advantage to AP is that it produces an extremely uniform coat even in complex part geometries due to the passive nature of the process. [11] This method is ideally suited for coating complex internal structures, assemblies and undercuts. Due to the release of ferrous ions, the coating permeates the porosity of the metal allowing for a more substantial and resilient bond between coating and surface material. In contrast to other coating processes, the base metal does not require a pretreatment with a phosphate coating in order to increase adhesion. In fact a phosphate pre-treatment may inhibit adhesion. The coating is non-toxic, produces little hazardous waste and has no fire hazard. The coating consists of pigmented water dispersible (latex) resin, hydrofluoric acid, hydrogen peroxide and deionized water. No solvents are used and coating has a low solids percentage. The average PH is 2.6 to 3.5. The final advantage of this process is that it is safe for workers due to the lack of required electrical current, noxious fumes and use of only mildly acidic chemicals. [1-6]

As recommended by the primary coating manufacturer, the Autophoretic process undergoes a four stage cleaning process. First, a one minute alkaline spray clean is employed to remove debris and remaining oils which prevent adhesion. This spray also neutralizes any acidity which may remain on the steel from prior operations. Next the component is immersed in an alkaline bath for two minutes, the bath serves the same purpose as the spray and is designed to equally expose all surfaces simultaneously. The component is then rinsed in plant tap water to remove excess alkaline followed by a final deionized wash to clean and deionize the component surface prior to coating. [1-4]

The components are then immersed in the coating tank at between 65°F and 70°F (18 to 21°C) for approximately 60 to 90 seconds to achieve desired coating thickness. Figures 1 through 3 below show the fundamentals of the coating process. [2-3]
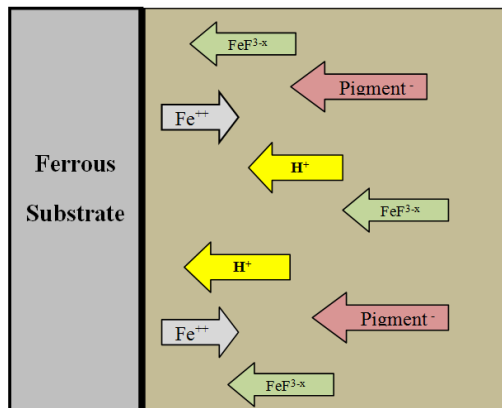

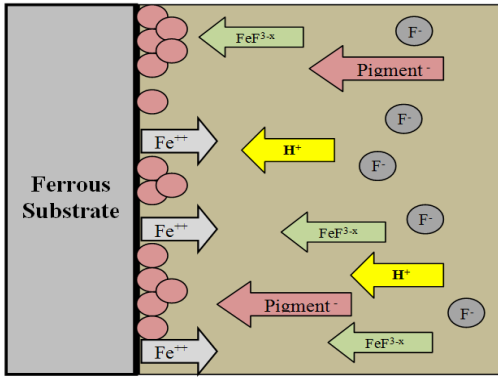**Figure1:** Stage 1 of Coating Process (Ion Transfer).

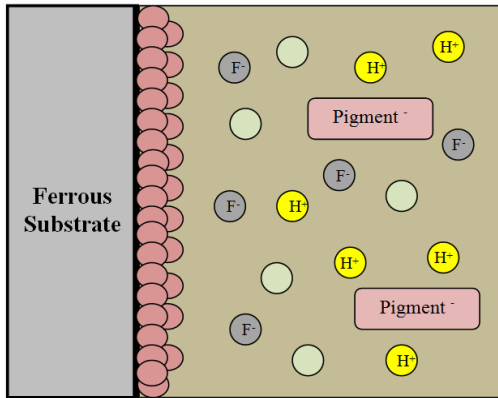**Figure2:** Stage 2 of Coating Process (Pigment Adherence)

Figure3: Stage 3 of Coating Process (Desired Coating Thickness Obtained and Part Removed)

Once coated the component is removed and allowed to air cure, known as flashing-off, before a rinse stage to allow the coating reaction to continue. The first rinse is an immersion in tap water followed by a second rinse in a non-chromate seal or DI water. The final step in the process is a heat cure in an oven at 210° to 356°F (99 to 180°C) depending on the resin type.

PROBLEM

Based on field observations, there has been questions as to whether Autophoretic (AP) coated components are providing the desired corrosion resistance properties. It is believed that either a flaw in the process, coating, or materials is resulting in premature failures in the field. Observable moderate to severe surface corrosion has been found with light coating applications of less than 0.5 mils (0.0127mm). This corrosion is more severe for parts experiencing wide cyclic climate changes, but has also been detected to a lesser degree for components in-service under climate controlled conditions. Additionally, both corrosion and coating delamination has occurred in thicknesses over 1.5 mils(0.0381mm). Minor to moderate corrosion has also been reported for components which were coated between 0.5 and 1.5 mills after six years in service in climate controlled conditions.

OBJECTIVE

As explained earlier, the coating bonds directly with iron in the metallic substrate. Therefore, the coating should theoretically provide a more complete and constant coating compared to alternative coating processes and thus provides a better barrier to corrosion. Accordingly, an investigation was conducted to determine the cause of the observed field failures, and to identify if the cause of corrosion is preventable.

Several hypotheses were made as to the cause of the corrosion failures:
1. Porosity of the coating
2. Erosion causing failure to occur
3. Acidity entrapment in the coating

Porosity was proposed as a potential issue due to the aqueous application of the coating. Excess moisture could become trapped within the coating and then off-gas during curing, creating porosity defects through the coating's surface.

Erosion and/or degradation of the coating were considered as a possible cause of corrosion due to spots/blemishes uniformly distributed over the surface of samples. It was believed that the coating was thinning over time and slowly perforating the coating. Additionally, a previous study suggested that microscopic blisters can form at delamination zones of the coating and therefore increase the corrosion rate [13]. In the study, the authors found the rate to be faster with AP coatings compared to cataphoretics and stated the cause was due to the absence of a pre-treatment layer.

Finally, excess acidity entrapment was considered. It was believed that acidity could potentially entrap between the base metal substrate and the coating material after curing. This would expedite corrosion or degrade the coating material to the point of failure.

It was hypothesized that porosity was the most likely cause of coating failure and therefore was evaluated first. A series of experiments were outlined, developed, and conducted to confirm or dismiss the presence of pores in the coating surface.

First, a Brauner, Emmet, and Teller (BET) test was used to determine the presence of micro-porosity of both AP and powder painted specimens. After that study was completed,

two forms of microscopy were to affirm the BET results as well as search for porosity too large to be detected by the BET experiment. Larger defects would be detected using between 10x and 100x optical magnification. Finer defects and micro-porosity were detected by using a Field Emission-Scanning Electron Microscope (FE-SEM). Finally, a comparative corrosion rate study was conducted to determine if AP coat is corroding at a faster rate than powder painted specimens.

METHODOLOGY

For the following experiments 12 gauge (0.1047 inch/ 2.657mm) sheet steel Q-Panels were used. All PVDC based Autophoretically coated specimens were coated in the same bath within a 5 minute period. All Powder Painted (PP) specimens were coated at the same time. All AP and PP panels were coated in the same facility on the same day. The PP panels along with an alternative epoxy-based AP coating were used for comparative analysis to the PVDC based coating. Finally, several identical Q-Panel specimens from a coatings manufacturer were provided. The panels were coated with an EPOXY based Autohporetic coating. These specimens were examined for comparison after the cause of failure was identified to determine if the cause persisted in the "Greener" AP option. These samples were not created at the same facility due to lack of equipment and material at that location.

Q-Panels were coated in a PVDC based Autophoretic material. The samples were coated on both sides of the panel with a single coat that ranged between 0.65 Mils (0.0065in./0.0165mm) and 0.72 Mils (0.0072in./0.0183mm) thick. The powder coated panels were coated with an epoxy based material on both sides with a single coat. The coating thickness ranged between 3.0Mils (0.003in./0.076) and 3.4 Mils (0.0034 in./0.0863mm). Coating thicknesses represented relatively standard thicknesses found in many industrial applications.

Multiple specimens were sent to an outside test facility to undergo the Society of Automotive Engineers (SAE) – Surface Vehicle Standard, J2334 Cosmetic Cyclical Corrosion Lab Test. Test specimens are placed in an enclosed chamber and exposed to a changing climate that comprises of the following three part repeating cycle. First specimens endure a 6.0 hour exposure to water (fog/condensing) humidity climate of 100%RH at +50C. This is followed by a 15 minute immersion in, or a direct spray of salt water at ambient temperature. This is followed by 17 hours 45 minutes of air drying in a climate of 50%RH at +60C. Multiple specimens were subjected to 10, 20, and 30 corrosion cycles.

BET Gas Absorption Test

The purpose of this study was to determine the size and distribution of molecular sized pores in AP and powder coated product. An abundance of porosity or large pore sizes could result in coating failure. The Brauner, Emmet and Teller (BET) gas absorption method was employed to determine the pore size and distribution of a specimen by determining the amount of gas which the specimen's surface absorbs. The gas condenses in the fine pore structure creating a layer on the specimen's surface which is used to find the surface area of the component. The gas pressure is increased until the surface pores of the specimen are saturated. The gas pressure is then released slowly evaporating the condensed gas from the system. Comparing the macroscopic surface area to the microscopic area, along with desorption of the isotherms, reveals information on the pore size, pore volume and pore area in a specimen.

First, a sample is subjected to a known gas at a known pressure. The sample site, or the surface of specimen, will begin to adsorb gas molecules at low pressure. As gas pressure increases, coverage of gas molecules increases to form a layer one molecule thick, the BET equation is then used to calculate the surface area of the specimen based upon the surface area of the gas. With the known surface area of the specimen the BET extrapolates the amount of pores in the surface. As the pressure continues to increase the gas continues to be absorbed by the pores until they are filled in. The pressure is released slowly allowing for pore volume to be determined based upon the overall volume of the gas that the surface absorbed. This entire process takes place in super cooled temperatures under vacuum on the samples. An example of how this how this process can be seen below in figure 4.

Stage 4: *Complete coverage and filling of pores. The BJH calculation can be used to determine pore diameter, volume and distribution.*

Stage 2: *Coverage of gas molecules increases with pressure to form a monolayer. BET equation is used to calculate surface area.*

Stage 3: *As gas pressure further increases, mulitple layers form. Smaller pores fill first.*

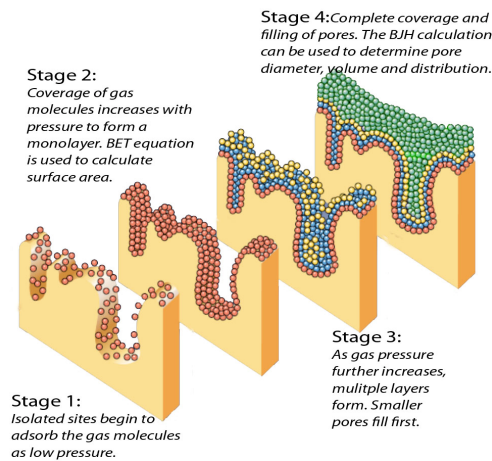Stage 1: *Isolated sites begin to adsorb the gas molecules as low pressure.*

Figure 4: BET Gas Absorption Process

For the BET experiment a Micromeritics Tristar 3000 with multi-gas capabilities was used. This particular machine restricts the size of samples to 0.25±0.01 inches in diameter(6.35±0.24mm). To increase the reliability 100 sample specimens were fit into each test tube to maximize surface area. A high accuracy punch of 0.25 ±.003 inches(6.35±0.076mm) in diameter was used to create 300 non-corroded specimens and 300 specimens which had been subjected to 30 corrosion cycles. Additionally 100 non-coated specimens were punched to be used as a control. Table 1 shows the experimental break down.

Table 1: BET Gas Absorption Process

BET Experiment: 1			
#	Sample Type	Samples/Tube	Surface Area [in2]
Tube 1:	AP- Non Corroded	100	18.1482 ± 0.5593
Tube 2:	AP- Non Corroded	100	18.1482 ± 0.5593
Tube 3:	Control	100	18.0406 ± 0.5571
BET Experiment: 2			
#	Sample Type	Samples/Tube	Surface Area [in2]
Tube 1:	AP- Non Corroded	100	18.1482 ± 0.5593
Tube 2:	AP- 30 cycle Corroded	100	18.5433 ± 0.5672
Tube 3:	Control	100	18.0406 ± 0.5571
BET Experiment: 3			
#	Sample Type	Samples/Tube	Surface Area [in2]
Tube 1:	AP- 30 cycle Corroded	100	18.5433 ± 0.5672
Tube 2:	AP- 30 cycle Corroded	100	18.5433 ± 0.5672
Tube 3:	Control	100	18.0406 ± 0.5571

Optical Microcroscopy

To analyze the surface of corroded and non-corroded specimens a micro-comparator was used. This study was conducted in the Western Michigan University Dimensional Metrology Laboratory. Three non-corroded Q-Panels would serve as a base line for the study. Observations would be taken from panels subjected to 10, 20, and 30 SAE-J2334 corrosion cycles. [10] Two corroded panels from each corrosion cycle would be studied. Additionally, one additional sample Q-Panel, created by an AP coating manufacture, would be used for comparison. This sample panel was created under ideal circumstances by a coating manufacture. All samples, except the manufacturer sample, were coated with PVDC Autophoretic coating and averaged the same 0.62 Mils thickness over its surface. The sample panel was coated with an epoxy based AP coating to an average thickness of 0.61 Mils thick.

Table 2: Optical Microscopy Testing Data

Optical Study Order				
	Corrosion Cycles	Base Material	Q-Panel Type	Coating
Sample 1:	Non Corroded	ASTM A1008	RS	PVDC
Sample 2:	Non Corroded	ASTM A1008	RS	PVDC
Sample 3:	10 Cycles	ASTM A1008	RS	PVDC
Sample 4:	10 Cycles	ASTM A1008	RS	PVDC
Sample 5:	20 Cycles	ASTM A1008	RS	PVDC
Sample 6:	20 Cycles	ASTM A1008	RS	PVDC
Sample 7:	30 Cycles	ASTM A1008	RS	PVDC
Sample 8:	30 Cycles	ASTM A1008	RS	PVDC
Sample 9:	Non Corroded	ASTM A1008	RS	Epoxy

Each specimen was inspected for large defects such as cracks, voids, scratches or any other breaches in the coating surface. The search for such defects was conducted at both 10x and 100x magnification. Specimens were examined for visible signs of corrosion formation. These studies focused on defect location and identification while attempting to determine if surface defects resulted in corrosion sites. Therefore size and quantity of corrosion or surface defects was not required. The testing order can be seen the table above (Table 2).

FE-SEM Study

To identify defects smaller than 10nm (3.937×10^{-7} in.), it was necessary to use higher magnification then the capabilities of the optical microscope. Therefore, a Field Emission-Scanning Electron Microscope (FE-SEM) was utilized. This microscope is housed at Western Michigan University's Biological Imaging Center. The laboratory features an ISI DS-130 scanning electron microscope with SIS Ultrascan 2 image acquisition software which is used to generate high quality digital images from the microscope.

Two 0.25±0.005 inch precision punched samples were created from one non-corroded PVDC AP Q-Panel. The same punch and procedure was used from the BET gas absorption section. The samples were affixed and layered with a 20nm thick layer of gold to prevent surface scattering caused by the PVDC material.

The primary objective of this study was to confirm the results of the BET gas absorption test and study defects found in the optical microscopy study.

Corrosion Rate

These sets of experiments were designed to accomplish two objectives. Firstly, these electrochemical tests determined if AP coat on manufactured in the sample facility as the other samples, corroded faster than Powder Paint. Secondly, this set of experiments determined if residual acidity was present in the AP material.

Corrosion occurs at a rate determined by equilibrium between opposing chemical reactions that take place on two

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dissimilar metals that are electrically connected, i.e. anode and cathode. The total current, the sum of anodic and cathodic currents, can be measured by sweeping the potential of the sample. Extrapolating the anodic and cathodic currents (which represent theoretical well-defined straight line current region) to an intersection, represents the corrosion current and corrosion potential. This is commonly known as a Tafel plot. The corrosion current is directly proportional to the corrosion rate, so a corrosion comparison can be made between samples by observing the corrosion current.

The working electrode (the samples), a reference electrode, and a platinum auxiliary electrode, and a 1 Molar sodium sulfide solution are used with an Electrochemical Workstation to obtain the Tafel plots. After the data was normalized to the sample surface area, Microsoft Excel™ was used to obtain the current intersection points.

RESULTS AND DISCUSSION

The following sections of this paper will discuss the results that were found in each of the various experiments.

BET Absorption

This series of tests found that the porosity in both PVDC AP and PP specimens were too small to be the primary cause of corrosion. The pore distribution was found to be smaller than the machines capability to record. Additionally, the pore sizes, that were found by the TriStar, were < 200µM. Therefore the diameter, depth, and distribution of the micro-pores that were detected, in both coatings, could not allow an electrolyte and/or oxygen access to the bare metal substrate to substantially affect the coating. Therefore this study suggests that microscopic pores do not compromise the coating.

Optical Microscopy

The optical microscopy revealed an abundance of surface defects on the A-Coat samples which would compromise coating integrity. These voids do not appear to be caused by the coating flaking, chipping, or general delaminating. They also do not appear to be cracks or pores left by off-gassing. The surface defects appear to be areas where the coating did not adhere to the metal substrate and left a void. The void areas appear at random in both placement and size. The one commonality of the voids was there unusual small size between specimens.

Under close inspection the voids expose a significant amount of substrate surface area and are present in all PVDC AP samples. Figure 5a below shows the surface of a non-corroded AP specimen as referenced, while Figure 5b shows the same image with the voids highlighted for comparison. It should be noted that even under ten times magnification, the highlighted voids are difficult to detect.
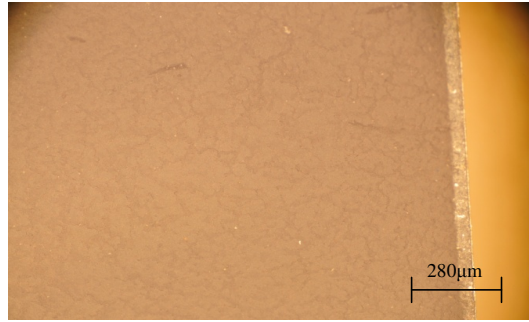

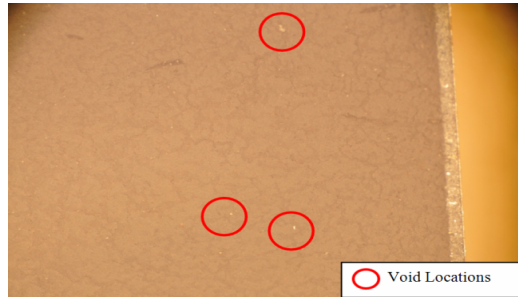**Figure 5a:** Non-corroded AP Sample (10x mag).

Figure 5b: NC-AP Sample Showing Voids (10x mag).

Figures 5a and 5b are meant to show that product could meet visual quality inspections while not properly coating product. Therefore it is plausible that manufacturers are releasing product which would not meet the productive needs of their consumers.

Some non-corroded specimens had an abundance of surface voids in isolated locations. Figure 6 below illustrates such an occasion where multiple voids are grouped together. This example also shows visible base metallic substrate and therefore has the potential for corrosion. The voids in Figure 6 have not been subjected to any corrosive cycles.
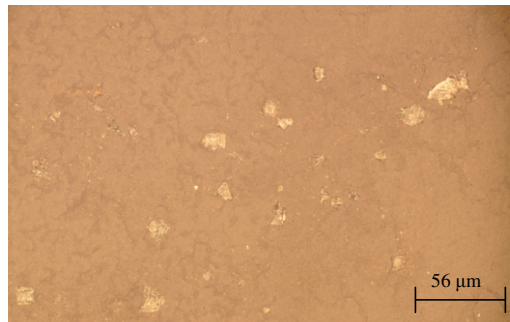

Figure 6: NC-AP Sample Showing Voids (50x mag).

Once discovered it became evident that these locations were the first sites of corrosion formation. Figure 7 below shows an AP sample that has been subjected to 10 corrosive cycles. The presence of corrosion, highlighted in blue, is visible inside several larger void locations.
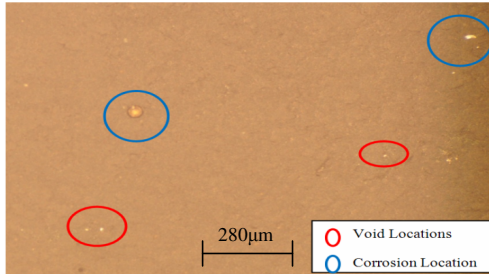

Figure 7: Corroded AP Sample (10x mag).

Using a side by side comparison (Figures 8 through 11) of the voids under 100x magnification it became evident that void locations begin to corrode around their periphery. Once the locations have filled in with corrosion they begin to flake the coating away. Figure 8a and 8b show void locations from AP coatings not subjected to the SAE-J2334 corrosion test. In these examples you can see the irregularity of the voids as well as the underlying non-corroded substrate.

Figure 8: NC-AP Sample Showing Voids (a right, b left).

Figure 9a and 9b show specimens subjected to 10 corrosion cycles. In both examples corrosion is visually evident. Figure 9a shows corrosion points beginning to form both in the interior as well as a light amount around the periphery. Figure 9b shows a void location with its perimeter fully encircled by corrosion.
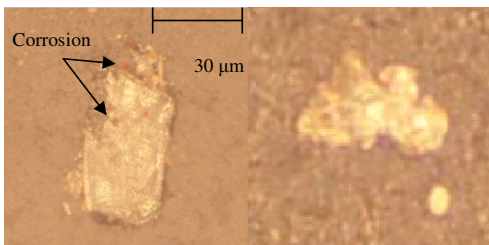
Figure 9: AP Sample Voids w/ 10 cycles (a right, b left).

Figure 10 illustrates AP voids which have been subjected to 20 corrosion cycles. With 20 cycles, visual rust becomes evident without microscopy and can be described as "Fly Spotting" hence the original porosity hypothesis. The majority of voids examined with 20 cycles are generally +50% filled with corrosion as seen in Figure 11b. Some voids were observed to also have begun to lift and further compromise the coating around the void periphery, as shown in 11a, without being fully corroded.
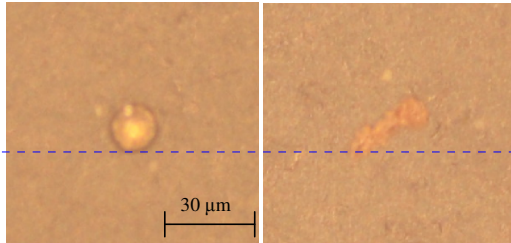

Figure 10: AP Sample Voids w/ 20 cycles (a right, b left).

Finally, Figure 11 shows void locations subjected to 30 corrosion cycles. All voids have been fully filled in by corrosion and have begun to fully compromise the coating. In most cases the sample's coating has begun to delaminate around the void. Figure 11a shows a void which fully corroded and has breached adjacent coating. Figure 11b shows a similar void once the breached coating has flaked.
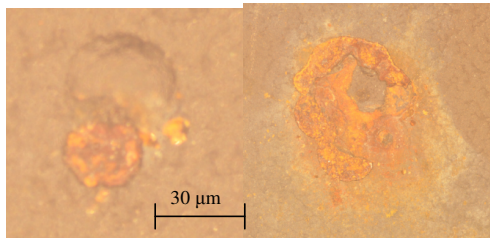

Figure 11: AP Sample Voids w/ 30 cycles (a right, b left).

Large visible rust spots and coating failure can be seen on specimens subjected to 30 cycles. In some isolated cases the corroded areas can reach as large as 0.25 inches in diameter.
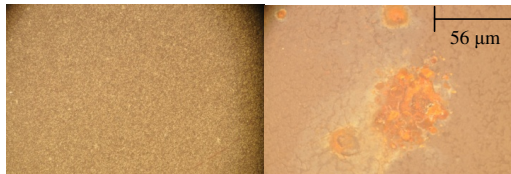

Figure 12: Corrosion w/ 30 cycles (PP left, AP right).

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As described earlier powder coated specimens were also subjected to the same SAE-J2334 cyclical corrosion test. Visual inspection showed that PP drastically out performed AP coated specimens. The visual surface corrosion found on AP samples with 10 cycles matched or exceeded PP which had been subjected to 30 cycles. See Figure 12 for details. Powder paint naturally leaves voids due to its application process. However, an Iron Phosphate wash is utilized in the pre-cleaning process to combat gaps in coverage. This same technique cannot be applied to AP due to Iron Phosphate contaminating the AP solution bath.

FE-SEM Study
The FE-SEM study confirmed that micro-porosity was not present in the AP coated specimens. Figure 13 shows an AP coated specimen at 5,000 µm resolution (100,000x). At this magnification several defects are present; specifically surface cracks from curing stress or degassing, micro-pores, as well as areas of dense or overlapping material.

These defects were studied under higher magnification to determine their origin and severity. Figure 13 shows one of the pore locations (highlighted in blue) in Figure 14. Under inspection it becomes evident that this pore does not penetrate the surface of the coating.
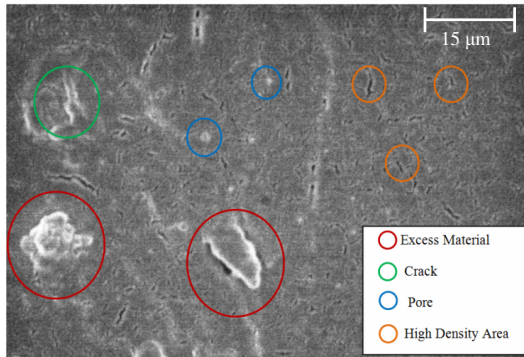
Figure 13: Non Corroded AP Surface under FE-SEM.
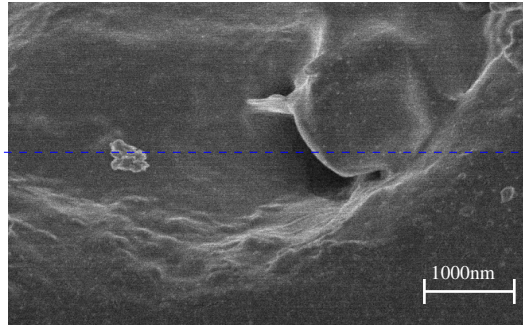
Figure 14: Surface Pore in AP Coating Sample.

Figure 15 is a higher magnification observation of a surface crack detected in Figure 13 (highlighted in green). As mentioned before this crack appears to have been caused either due to stress of the curing process or by off-gassing. Although it is indeterminate whether this crack fully penetrates the surface of the coating it should be noted that this crack is as fine as a human hair.   Therefore the likelihood of this crack resulting in accelerated corrosion, greater than the void location, is very small.
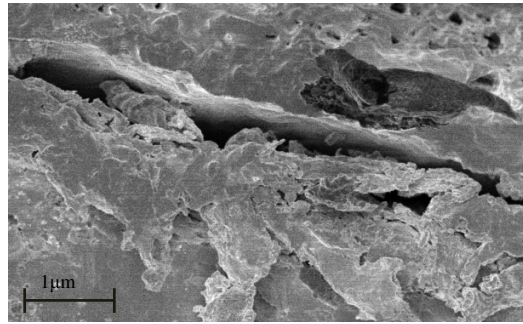

Figure 15: Surface Micro-Crack in AP Coating Surface.

To better understand the void areas the perimeter of one such void was also inspected under FE-SEM. Figure 16 depicts the boundary of a void. The top surface of the coating is denoted in the upper right corner while the base metal, although out of sight, would be located in the lower left corner. Review of the void around the periphery showed formation of microscopic iron oxide deposits (highlighted in green). It should be noted that this specimen was not subjected to any corrosion tests.
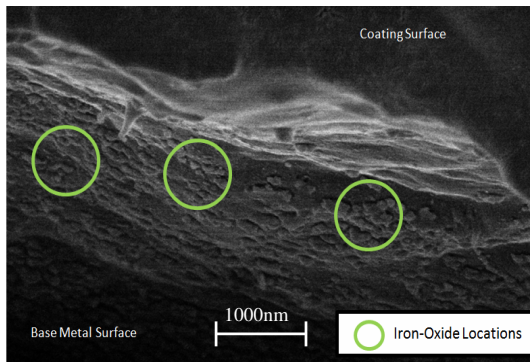
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Figure 16: Surface Void Periphery Boundary.

Corrosion Rate

The results obtained from the Tafel plots indicate AP coatings have a higher corrosion current density than powder paint coatings, which insinuates a higher corrosion rate. Consistent Tafel plots for each sample were obtained, and anodic/cathodic currents could be extrapolated to find the corrosion current. Figures 17 and 18 below show a comparison of bare, AP and powder paint coated sample Tafel plots and corrosion current, respectively. It should be noted that Figure 18 shows a distinctive difference in current density between powder paint and AP. However, it was also determined that the AP samples had a higher corrosion density then that of the bare metal control sample which was used. This phenomenon may have been caused due to breaches in the coating which expedite corrosion.
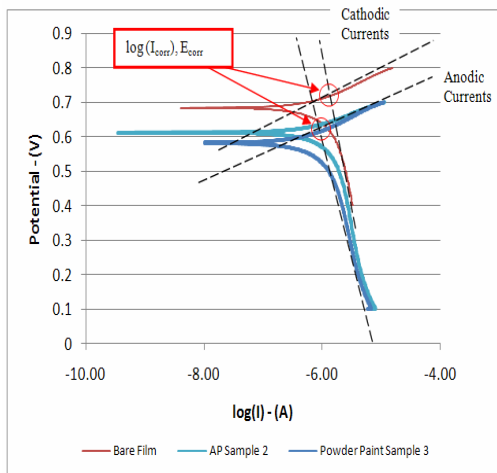
Figure 17: Tafel Plot
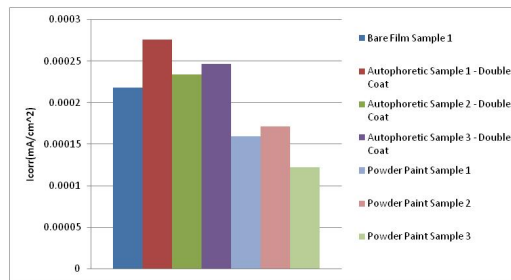
Figure 18: Corrosion Rate Study

CONCLUSIONS AND RECOMMENDATIONS

Porosity in the coating surface, at both the macro and micro scale, are too small and few to have a significant impact on corrosion. Instead coating voids in the surface of Autophoretically coated materials are the primary locations of corrosion. An obvious progressive corrosion pattern is evident starting at locations which were improperly or inadequately coated. It has been observed that these defects cause a systemic problem which compromises the overall coating surface.

Oil residue is known to cause similar surface voids on a macroscopic scale. It is possible that the voids are the result of microscopic oil particles which were improperly cleaned from the surface of the substrate. Additional testing should be conducted to determine if advanced cleaning techniques could reduce or eliminate this phenomenon

Autophoretically coated steel panels have a higher corrosion potential then Powder Painted panels. This could be directly tied to the surface defects found in the panels. If the defects were eliminated there is the possibility that AP could approach or match powder paint's corrosion potential.

ACKNOWLEDGMENTS

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