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Alkaline Corrosion-Resistant Sealant for Anodized **Aluminum Alloys**

ABSTRACT

Corrosion resistance of anodized aluminum sealed with conventional and a new sealant was tested against the alkaline, acid and neutral chloride salt solutions. The results show that the conventional water based sealants have good acid and Neutral Salt Fog resistance; however, they do not possess the necessary alkaline corrosion resistance to be used for decorative and automotive exterior applications. A new anodic coating sealant was developed to provide the necessary alkaline corrosion resistance along with the acid and neutral salt attack resistance. Acid Dissolution Test (ADT) Rating, Alkaline Corrosion Resistance, Neutral Salt Fog, Heat Resistance and Alkaline Car Wash Detergent tests were done in accordance with General Motors Spec GMW 14665. Results revealed that this novel sealant is significantly capable of meeting the rigorous GMW 14665 specification and can be used for sealing the anodic coatings used in automotive exterior applications.

nodizing is an efficient electro-Achemical conversion coating method to improve the corrosion and wear resistance of aluminum alloys. In general, acid, alkaline and salt attack resistance of the anodic films depends on the integrity, physicochemical properties, thickness, and the sealant type used as a post-treatment for anodic coatings [1,2]. Sealants improve the corrosion resistance of anodized aluminum by closing the micro-pores of anodic films to prevent the corrosive media from reaching the aluminum substrate [2]. Hydrothermal, nickel acetate, and dichromate are the most commonly used commercially available anodized aluminum sealants. Cold nickel fluoride and trivalent chromium-based room temperature anodized aluminum sealants are relatively new technologies.

All of these commercial sealants have the advantage of being water based and exhibit relatively good acid and salt attack resistances. However, they do not have the necessary alkaline corrosion resistance to be used for decorative and automotive exterior applications above beltline per GMW 14665 [3].

In the present study, the alkaline, acid, and neutral salt spray resistance of anodic coatings sealed with the new MLT anodizing sealant was investigated. A comparative performance study between the commercially available seals and this newly developed MLT anodic coating sealant was made.

1 EXPERIMENTAL.

6061-T6 wrought aluminum alloys were used for experiments. Test coupons were degreased and cleaned in alkaline cleaner. Cleaned and degreased coupons were rinsed in flowing tap water for 1 min. After alkaline etching and rinsing, coupons were deoxidized in mixed acids at room temperature and rinsed in flowing tap water for 1 min. Thin film sulfuric acid anodizing (12 ASF (1.3 A/dm²), 7.5 mm) was performed in an electrolyte consisting of 190 g/L sulfuric acid and 7 g/L Al³⁺ ions at 21°C. The processing details of the applied post treatment processes for the anodized aluminum samples are listed in **Table 1**. The resultant anodic coating thickness was measured in accordance with ASTM B244 [4] using a precalibrated eddy current instrument.

High pH alkaline corrosion resistance of the sealed anodized aluminum was measured by immersing the test coupons in a high pH test solution prepared in accordance with GMW 14665 [3]. The measured pH of the test solution was 13.0 and the immersion time was 10 min at room temperature. Alkaline corrosion resistance tests were performed, both without, and after exposure to car wash abrasion testing. The sealed sample was continuously scrubbed with a nylon bristle brush and car

POSTTREATMENT	CHEMICAL SOLUTION	TEMPERATURE (°C)	TIME (MIN)
Hydrothermal	DI water + 0.5% v/v METALAST Seal 6000	100	30
Nickel Acetate	4% v/v METALAST Seal 6100	88	20
Trivalent Chromium	25% v/v Trivalent chromium (TCP)	21	5
New MLT Sealant	Water based - No heavy metal	21	3-10
Table 1. Post Treatment Processes for Anodized Aluminum			

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wash detergent solution up to 200 motions back and forth prior to the alkaline resistance test. Sealed samples with no surface change after the alkaline immersion test were exposed to Neutral Salt Fog test for 480 hours in a salt spray chamber maintained in accordance with ASTM B117 [5]. Alkaline car wash detergent resistance was measured in a modified car wash detergent solution, described in GMW 14665, with a pH of 11.2. Test coupons were immersed in room temperature detergent solution for 10 min to observe any surface change.

Temperature resistance of the sealed anodic coatings was measured after heating the test coupons at 90°C for 24 hours in air, and the evaluation was done while the test coupons were still hot. Acid Dissolution Test (ADT) rating was calculated per GMW 14665 after measuring the coating weight dissolved in chromic-phosphoric acid solution as per ASTM B680 [6] and the total coating mass in mg as per ASTM B137 [7]. Dye Stain test on sealed anodized aluminum samples was done in accordance with ASTM B136 [8].

Water contact angle measurements were carried out to quantify the degree of hydrophilicity using a Rame-Hart model 250-F1 contact angle goniometer.

Field Emission Scanning Electron Microscopy (FE-SEM) images of the anodized aluminum before and after sealing were acquired with a Hitachi S-4700 FE-SEM.

Wet tape adhesion studies were performed in accordance with FED-STD-141, Method 6301 [9]. Sealed anodic coatings were primed with a non-chromated water based epoxy primer qualified to MIL-PRF-23377J (Type I, Class N) [10].

2 RESULTS AND DISCUSSION

2.1 Alkaline and Neutral Salt Fog Corrosion Resistance. High pH alkaline corrosion performance and dye stain



Figure 1. Alkaline corrosion resistance (pH = 13) per GMW 14665 and Dye Stain Test per ASTM B136 results for anodized aluminum post-treated with the conventional sealants. H: Hydrothermal, HT: Duplex Hydrothermal and Trivalent Cr, T: Trivalent Cr, and NT: Duplex Nickel Acetate and Trivalent Cr.

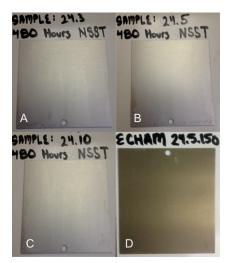
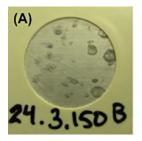
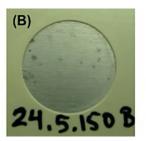


Figure 2. Alkaline corrosion resistance (pH = 13) per GMW 14665 and Dye Stain Test per ASTM B136 results for anodized aluminum post-treated with the new MLT sealant for 3 min (A), 5 min (B), and 10 min (C) at 65 $^{\circ}$ C for clear anodized aluminum and 5 min MLT sealant after electrolytic coloring (D).





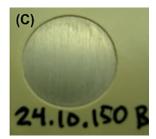


Figure 3. MLT sealed anodized aluminum after being exposed to high pH alkaline test solution for 4ks. Samples were sealed for 3 min (A), 5 min (B), and 10 min (C) at 65°C.

test (ASTM B136) results for the traditional sealants, including hydrothermal, duplex hydrothermal and trivalent Cr, trivalent Cr, and duplex nickel acetate and trivalent chromium seals are shown in Figure 1. All panels were anodized to 7.5 µm, and the operating parameters for the sealants were given in **Table 1**. The dip line indicates that commercially available traditional sealants were attacked by the high pH alkaline test solution. On the other hand, dye stain test was done above the dip line and conventionally sealed samples passed the test with no stain formation. Samples post-treated with a clear organic coating provided the necessary alkaline corrosion and 480 hours Neutral Salt Fog resistance with no demarcation and/or pit formation. However, additional coatings or topcoats before or after sealing are not a robust and economical way to provide the necessary alkaline corrosion resistance on anodized aluminum. Furthermore, using additional topcoats such as organic or inorganic paints, sol-gel, nanoparticles, physical or chemical vapor deposits, etc. will not meet the requirements of GMW 14665 specification.

Figure 2 shows the MLT sealed test coupons after high pH alkaline immersion test followed by 480 hours salt spray. Anodized test coupons sealed with MLT sealant for 3 min, 5 min, and 10 min at 65 °C prevented the alkaline corrosion and neutral salt fog attack. Similarly, two-step electrolytic colored anodized aluminum (20 μm) sealed with new MLT sealant provided the necessary alkaline and neutral salt fog resistance to meet the GMW 14665 requirements. In addition, MLT sealed samples did not

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