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A Comparison of Alternatives to Chromic Acid Anodizing

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ABSTRACT

Chromic acid anodizing (CAA) is an aluminum surface pretreatment currently used on military aircraft and equipment. Chromium VI, present in this process, is a carcinogen and federal, state and local environmental agencies have implemented legislation which restricts the use of this material. The Naval Air Warfare Center Aircraft Division Warminster investigated several alternatives to CAA including: Sulfuric-Boric Acid Anodize, Sulfuric Acid Anodizing, and Phosphoric Acid Anodizing. Physical performance properties of these processes were characterized and as a result of this program, the MIL-A-8625 anodize specification was modified to include some of these alternatives.

INTRODUCTION

Chromic acid anodizing (CAA) is a common surface pretreatment for aluminum currently used on Navy aircraft, weapon systems and ground support equipment. This anodize process forms a thick oxide film which provides protection against chemical degradation from the operational environment. MIL-A-8625F "Anodic Coatings, for Aluminum and Al Alloys" Type I covers the performance requirements of CAA. While this anodize process offers satisfactory performance, it contains Chromium VI which is a carcinogen. Recently, California's South Coast Air Quality Management District's (AQMD) Rule #1169 governing the hazardous emissions limits for electrolytic chromium processes went into effect. A national regulation on emissions from chromium electroplating operations was also issued by the Environmental Protection Agency under the Clean Air Act Amendment of 1990 with limits similar to the SCAQMD Rule #1169. Finally, the Department of Defense (DoD) has determined that Chromium is one of the major components in the waste generated by its maintenance depots and operations (Ref 1). Therefore, Chromium must be eliminated or minimized from the emissions of this process.

Two approaches are available to attain this goal. One approach is through the incorporation of process emission controls. The other approach is to eliminate the source of the hazardous material (i.e. CAA) through the use of alternative technologies. While both of these methods reduce the amount of hazardous material released, the latter also solves Chromium disposal and handling concerns and eliminates the need for expensive control equipment required by 1994 under current AQMD laws. Control equipment for each Navy Depot has been initially estimated at $500K-1,000K for capital costs and $250K-600K for annual operating and disposal costs. An adequate replacement to provide protection against environmental degradation is particularly important to the Navy, considering the severe environment in which it operates, as well as the cost of the aircraft, weapon systems and ground support equipment. The Naval Air Warfare Center Aircraft Division at Warminster (NAWCADWAR) has an extensive environmental materials program aimed at the elimination of hazardous materials from Navy aerospace processes (Ref 2 & 3). An effort under these programs was established to evaluate alternative technologies to CAA. The following is a description of this program.

DESCRIPTION OF PREPARATION AND ANODIZE PROCESSES

Surface preparation is an essential step in the process of forming protective pretreatments for aluminum. Surface preparation consists of several steps: cleaning, etching (optional) and deoxidizing. Alkaline cleaners, etchants and deoxidizers were used to remove organic contaminates and any remaining surface oxides prior to chemical treating. The materials used in the preparation of the test specimens were non-silicated, non-chromated alternatives which are described in reference 4. The CAA control process used in this investigation is covered by MIL-A-8625F Type I. Specific details on CAA of aluminum are provided in references 5-6.

Several potential alternatives have been identified for replacement of chromic acid anodizing. These alternatives are: Sulfuric Acid Anodize (SAA), Sulfuric Boric Acid Anodize (SBAA), and Phosphoric Acid Anodize (PAA). Descriptions of these processes are provided in References 6 - 10. Thin Film Sulfuric Acid Anodizing is another alternative currently being investigated and will be reported on at a later date. To evaluate these alternatives, laboratory scale process lines were set up at NAWCADWAR. These operations were used to analyze the performance properties of the different anodizing processes in comparison to CAA, both sealed and unsealed on various substrates. These films were examined as pretreatments for standard Navy coatings using the procedures described below. The results from this evaluation were used to determine the effectiveness of these non-chrome alternatives to provide equivalent corrosion resistance and paint adhesion to that provided by CAA. The fatigue characteristics of these processes were not evaluated as part of this study and would have to be evaluated prior to use of any of these alternatives. A previous study examined the fatigue properties of SBAA and found them to be comparable to CAA (Ref 12).

EXPERIMENTAL

The performance properties of the anodize processes were evaluated on common aluminum alloys and with standard Navy coating systems. Physical performance tests (i.e. bare and painted corrosion resistance, coating adhesion, coating weights, etc.) were used to evaluate the anodize films. The following is a description of the substrates, coatings, and experimental procedures used in this investigation.

Materials - The substrates used in this study were bare 2024 T-3 and 7075 T-6 aluminum alloys. Table 1 lists the coatings applied to these substrates in this investigation. Sets of test specimens were prepared at NAWCADWAR following the manufacturers' recommended procedures. A non-silicated, non-chromated alkaline cleaner and non-chromated deoxidizer were used in the preparation of all specimens (Turco's 4215-NC-LT and Smut-Go-NCB products, respectively).

Anodize Seals - The chromic and sulfuric acid anodized specimens were sealed using the standard 5% dichromate seal at 93°C for 15 minutes as specified in MIL-A-8625E. The sulfuric/boric and thin film sulfuric acid anodize specimens were sealed with a dilute chromic acid seal which is described in reference 7. The Phosphoric Acid Anodize specimens were not sealed.

Coating Weight Determination - Anodize film coating weights were obtained using the test procedure outlined in MIL-A-8625E. Weights for sealed and unsealed films were determined on the test alloys. The weights were recorded in mg/ft^2.

Adhesion and Water Resistance - Adhesion of organic coating systems to the anodize films was evaluated using two methods: wet tape adhesion and scrape adhesion. The wet tape test is a modified version of the American Society for Testing and Materials ASTM D 3359, method A. This test was performed by immersing a specimen in distilled water for a period of time at a specific temperature. Three immersion conditions were used for this test: 24 hours at 23°C, 96 hours at 49°C, and 168 hours at 65°C. Upon removal, two parallel scribes, 3/4 inch apart, were cut through the coating and into the substrate. An "X" was subsequently scribed through the coating between the two initial scribes. A strip of 3M 250 masking tape was applied firmly to the coating surface perpendicular to the scribe lines and immediately removed with one quick motion. The specimens were examined for removal and uplifting of the coating from the substrate and the adhesion rating was recorded. Table 2 gives the performance description for these adhesion ratings. In addition, the water resistance of the pretreatment and coating systems was characterized by examining the test panels for softening, uplifting, blistering, and other coating defects and substrate corrosion which may have resulted from the exposure.

The scrape test was performed in accordance with ASTM D 2197, method A on specimens with a section of the substrate surface exposed. The instrument used to perform this test was a SG-1605 Scrape Adhesion Test Apparatus manufactured by Gardner Laboratory. The test was performed by guiding a weighted stylus at a 45° angle to the specimen along the exposed substrate into the coating system. The scrape adhesion was recorded as the heaviest weight used without shearing the coating from the substrate.

Corrosion Resistance - Five aluminum specimens 3"x10" of each anodize process were exposed in 5% salt spray (ASTM B 117) for 336 hours. Upon removal, the panels were inspected for evidence of corrosion. In addition, four aluminum specimens of each unsealed anodize film/coating system were scribed with a figure

"X" through the coating into the substrate. Two specimens were exposed in 5% salt spray (ASTM B 117) for 2000 hours and two were exposed in SO_2/salt spray (ASTM G 85) for 500 hours. The panels were then inspected for corrosion in the scribe area and blistering of the coating. Subsequently, one panel from each exposure was chemically treated to remove the organic coating without disturbing the substrate and the specimen was examined for corrosion.

RESULTS AND DISCUSSION

Test panels were processed with non-chromate cleaners and deoxidizers, and then anodized with the subject processes. Coating adhesion, water resistance, & corrosion tests were performed using MIL-P-23377 epoxy primer, MIL-P-85582 epoxy/waterborne primer, and TT-P-2760 polyurethane/elastomeric primer. Also, specimens primed with MIL-P-23377 and topcoated with MIL-C-85285 high solids polyurethane were tested. These coatings are described in references 13-16. The following is a summary and discussion of the results.

Table 3 shows coating weights for the anodize processes on the two different aluminum alloys. Coating weight gives an indication of oxide film thickness and is determined by processing variables such as amps/ft^2, time, etc. Both sealed and unsealed weights were obtained for each process except for PAA. Sealed PAA specimens were not evaluated since the morphology of PAA is not the same as the other anodize films and sealing is not applicable to this process. The relative coating weights for unsealed specimens ranged from 40 mg/ft^2 for PAA, to 300-600 mg/ft^2 for CAA & SBAA, to greater than 1000 mg/ft^2 for SAA.

Enhanced coating adhesion is one of the primary function of a surface pretreatment. These coating adhesion tests were performed on unsealed anodize films immediately after the 7 day cure time for the coatings. With further aging of the finishing system, adhesion normally improves, so these results are considered the minimum values. The results of the adhesion/water resistance tests are provided in Tables 4 to 6.

The results from the 24 hour tape tests performed on these processes showed adhesion values of 5A (one 4A result) for all coating systems tested (Table 4). These results indicate virtually no susceptibility to coating-substrate disbondment upon exposure to water. Most aerospace coatings use this adhesion requirement. In the expanded adhesion tests (4 & 7 day), all of the anodize processes, with various coating systems, continued to exhibit good to excellent adhesion & water resistance (Tables 5 & 6). The SBAA and CAA processes were consistently the best performing. This is evidenced by the tape test 5A results after extended immersion in water.

A standard aerospace requirement for scrape adhesion is 3 kg. The overall scrape adhesion results for all processes tested ranged from 0.5 kg to 10.5 kg. (Table 7). This indicated that other factors (such as the coating edge effects, pretreatment thickness, pre-paint surface cleanliness, etc.) affected the outcome of the tests. The results from the sulfuric/boric acid anodized process ranged from 2.0 kg to 7.0 kg. and was the process most comparable to chromic acid anodizing.

Sealed, unpainted specimens from all of the processes were exposed to 5% salt spray (ASTM B117) on 6° racks and examined at 24 hour intervals for evidence of corrosion. Total exposure time was 336 hours. These results are summarized in Table 8. The PAA panels failed in less than 72 hours indicating poor bare corrosion resistance. This is not unexpected considering the stalagmite structure of this oxide. All of the remaining anodic processes passed 336 hours of exposure on all alloy specimens without any evidence of surface corrosion, indicating excellent anodic coating performance.

Corrosion resistance is an important property for Navy aircraft coatings due to the severe operational environment in which the aircraft are deployed. Therefore, most aircraft primer specifications have a minimum of 1000 hours exposure to salt spray as the corrosion resistance requirement. The anodic coating plays an integral role in meeting this requirement by maintaining the integrity of the coating/substrate interface. To evaluate this property, painted specimens for all anodize processes were exposed to 5% salt spray (ASTM B117) and examined for corrosion in the scribe area and blistering of the coating. These results are summarized in Tables 9 & 10. All of the anodize processes, with all coating systems, passed 1000 hours of exposure. There was little to no corrosion products in the scribe and no blistering of any of the coatings.

Since all processes performed well for over 1000 hours on both substrates, the test was continued for another 2500 hours. At 3500 hours, there was still virtually no corrosion products in the scribe or blistering of the coating on any of the specimens. Subsequently, the coatings were carefully removed from the surface with a chemical stripper, without disturbing the underlying substrate. Upon further examination, there was no evidence of underlying corrosion on these panels. At 3500 hours, while all of the primed and primed/topcoated specimens performed well, the SBAA and CAA specimens showed the least amount of corrosion products in the scribe area.

Painted specimens exposed to SO_2/salt spray (ASTM G85) were also examined for damage to the coating and corrosion in and away from the scribe, and these results are summarized in Tables 11 & 12. The SO_2/salt spray environment simulates industrial exhaust gases, such as those found on aircraft carriers from engine exhausts, and it is an extremely aggressive environment. Most aircraft coating specifications do not have exposure to SO_2/salt spray as a corrosion resistance requirement. Therefore, the exposure periods selected were based on differences in finishing system performance.

Primed panels, after being exposed for 168 hours, were examined for signs of corrosion. As seen in Table 11, the results for this test were mixed between the alloys, primers and anodize films. In these tests, the SBAA appeared to show the weakest performance. However, the diversity of these results (from passing to complete failure) made drawing specific conclusions difficult and is partially the reason why coating specifications do not call out this test. The primed and topcoated 7075 specimens all failed between 168 hours and 336 hours. The primed and topcoated 2024 specimens were borderline failures at 500 hours, except for the CAA which failed at 336 hours. On both alloys, the CAA process performed the worst of all those tested.

In general, the corrosion resistance of several of the anodize processes, in combination with the standard epoxy primer or the epoxy primer/polyurethane topcoat coating systems, was equivalent to the performance of the chromic acid anodize controls. This equivalent performance for these non-chromated anodic coatings as compared to the chromated anodic coating is due to a high degree of interfacial integrity between the coating and substrate.

SUMMARY

The goal of this effort was to evaluate non-chromated alternatives for chromic acid anodizing used on current aerospace structures. The results from this evaluation show that some of the alternative anodize processes have comparable performance properties to CAA. The SBAA and SAA test results show that they provide acceptable performance for corrosion resistance and coating adhesion. The coated PAA specimens performed fairly well, however, the unpainted specimens failed rapidly in the corrosion tests. As stated earlier in the report, the fatigue characteristics of these processes were not evaluated and may be an issue for the use of these materials. For example, Sulfuric Acid Anodize has traditionally been considered to be a fatigue sensitive process and should not be used in fatigue critical areas. The MIL-A-8625 military specification, which covers CAA and SAA, has been revised to include the SBAA alternative. In 1993, the SBAA was authorized by the Naval Air Systems Command as an alternative to CAA. The use of a non-chromated process will allow the Navy to meet stringent environmental standards while maintaining operational readiness and efficiency of system performance. In addition, significant cost savings ($M) will be recognized by avoiding the need to implement emission control equipment.

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TABLE 1: ORGANIC COATING SYSTEMS EVALUATED*

MIL-SPEC	TYPE	FUNCTION	THICKNESS
MIL-P-23377D	1	Epoxy Primer	0.6-0.9 mils
MIL-P-85582A	1	Epoxy Primer	0.6-0.9 mils
TT-P-2760	1	Polyurethane Primer	0.8-1.2 mils
MIL-P-23377D	1	Epoxy Primer	0.6-0.9 mils
MIL-C-85285	1	Polyurethane Topcoat	1.8-2.2 mils

* All coatings were applied by conventional air spray & allowed to cure for seven days prior to testing.

TABLE 2. ASTM D3359 ADHESION RATINGS

Rating		Description
5A	-	No peeling or removal
4A	-	Trace peeling or removal along incisions
3A	-	Jagged removal along incisions up to 1/16 in. (1.6 mm) on either side
2A	-	Jagged removal along most of incisions up to 1/8 in. (3.2 mm) on either side
1A	-	Removal from most of the area of the X under the tape
0A	-	Removal beyond the area of the X

TABLE 3. COATING WEIGHT TEST RESULTS - (mg/ft^2)

	ANODIZE PROCESSES			
ALLOY	PAA	SBAA	CAA	SAA
2024-T3 Sealed	---	150.9	576.4	1115.2
7075-T6 Sealed	---	429.9	567.2	1731.2
2024-T3 Unsealed	40.3	372.8	416.0	904.3
7075-T6 Unsealed	43.2	576.0	315.5	1397.9

TABLE 4. 24 HOUR ADHESION/WATER RESISTANCE TEST RESULTS

		ANODIZE PROCESSES		
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	5A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	4A
7075 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	5A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	5A

TABLE 5. 4 DAY ADHESION/WATER RESISTANCE TEST RESULTS

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	3A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	5A
7075 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	3A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	4A

TABLE 6. 7 DAY ADHESION/WATER RESISTANCE TEST RESULTS

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	4A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	4A
7075 - MIL-P-23377	5A	5A	5A	5A
- MIL-P-85582	5A	5A	5A	5A
- TT-P-2760	5A	5A	5A	5A
- MIL-P-23377/ MIL-C-85285	5A	5A	5A	5A

TABLE 7. SCRAPE ADHESION TEST RESULTS - (kg)

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	0.5	3.0	6.5	1.5
- MIL-P-85582	7.0	3.5	9.0	3.0
- TT-P-2760	1.5	3.0	3.5	4.0
- MIL-P-23377/ MIL-C-85285	6.0	4.0	6.0	10.5
7075 - MIL-P-23377	2.0	7.0	5.0	4.5
- MIL-P-85582	6.0	4.5	7.0	5.5
- TT-P-2760	1.5	2.0	3.0	1.5
- MIL-P-23377/ MIL-C-85285	4.0	3.5	6.0	3.0

TABLE 8. 5% SALT SPRAY RESULTS - 336 HRS (BARE PANELS)

	ANODIZE PROCESSES			
ALLOY	PAA	SBAA	CAA	SAA
2024-T3 Sealed	FAIL*	PASS	PASS	PASS
7075-T6 Sealed	FAIL*	PASS	PASS	PASS

(* Failure occurred at less than 72 hours of exposure)

TABLE 9. 5% SALT SPRAY TEST RESULTS (1000 HRS)

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	PASS	PASS	PASS	PASS
- MIL-P-85582	PASS	PASS	PASS	PASS
- TT-P-2760	PASS	PASS	PASS	PASS
- MIL-P-23377/ MIL-C-85285	PASS	PASS	PASS	PASS
7075 - MIL-P-23377	PASS	PASS	PASS	PASS
- MIL-P-85582	PASS	PASS	PASS	PASS
- TT-P-2760	PASS	PASS	PASS	PASS
- MIL-P-23377/ MIL-C-85285	PASS	PASS	PASS	PASS

TABLE 10. 5% SALT SPRAY TEST RESULTS (3500 HRS)

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	PASS	PASS	PASS	PASS
- MIL-P-85582	PASS	PASS	PASS	PASS
- TT-P-2760	PASS	PASS	PASS	PASS
- MIL-P-23377/ MIL-C-85285	PASS	PASS	PASS	PASS
7075 - MIL-P-23377	PASS	PASS	PASS	PASS
- MIL-P-85582	PASS	PASS	PASS	PASS
- TT-P-2760	PASS	PASS	PASS	PASS
- MIL-P-23377/ MIL-C-85285	PASS	PASS	PASS	PASS

TABLE 11. SO_2/SALT SPRAY TEST RESULTS (168 HRS)

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377	P	F @108	P	P
- MIL-P-85582	P	F @168	F @168	P
- TT-P-2760	F @108	F @48	F @168	F @108
7075 - MIL-P-23377	P	F @108	-	P
- MIL-P-85582	F @168	F @168	+	F @108
- TT-P-2760	F @108	F @48	P	F @48

(P = Pass, + = Borderline Pass, - = Borderline Failure and F @### = Failure at # Hours)

TABLE 12. SO_2/SALT SPRAY TEST RESULTS (336 HRS)

	ANODIZE PROCESSES			
ALLOY (Unsealed)	PAA	SBAA	CAA	SAA
2024 - MIL-P-23377/ MIL-C-85285	-	-	F @336	-
7075 - MIL-P-23377/ MIL-C-85285	F @336	F @250	F @168	F @168