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A COMPARISON OF SULFURIC ACID/BORIC ACID ANODIZE AND CHROMIC ACID ANODIZE PROCESSES

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Chromic acid anodizing (CAA) is an aluminum surface pretreatment currently used on Navy aircraft and weapon systems. This anodize process forms a thick oxide film which provides protection against environmental degradation. This is particularly important due to the severely corrosive naval aviation operational environment. Chromium VI, however, is a carcinogen and its wide spread use as a corrosion inhibitor in this process is being restricted. The Navy has targeted chromated maintenance operations for reduction of hazardous waste generation. Several alternative candidates have been identified: Phosphoric Acid Anodizing, Boeing Aerospace Corp.'s Boric-Sulfuric Acid Anodize (SBAA) and thin film sulfuric acid anodizing. The Boeing SBAA process was selected for optimization and service demonstration. The Naval Air Warfare Center Aircraft Division Warminster analyzed the performance properties of SBAA and CAA both sealed and unsealed on various substrates with and without standard Navy coating

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systems. The results of this program show that SBAA effectively provides equivalent corrosion resistance and paint adhesion while maintaining the existing mechanical properties provided by CAA. Replacement of CAA eliminates the need for expensive control equipment required by 1994 under current AQMD laws, resulting in $M's in cost avoidance for the Navy.

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INTRODUCTION

With the recent increase in environmental awareness, federal, state and local environmental agencies (EPA, California's Air Quality Management Districts (AQMD), etc.) have issued legislation governing the handling, use and disposal of hazardous materials and wastes. National regulations such as the Clean Air and Water Acts, Resource Conservation and Recovery Act, and local rules like California's South Coast AQMD Rule #1169 limit or prohibit the hazardous emissions generated from the use of these materials. The Department of Defense (DoD) has determined that the majority of hazardous materials and hazardous waste generated by the DOD comes from its maintenance depots and operations (Ref 1). The bulk of these hazardous compounds are associated with cleaning, pretreating, plating, painting and paint removal processes. Chromium is one of the major components in the waste generated from these processes. A national regulation on emissions from chromium electroplating operations is being pursued by the Environmental Protection Agency (Ref 2) with limits similar to the SCAQMD Rule #1169. Chromic acid anodizing is a common surface pretreatment for aluminum, which is currently used on Navy aircraft, weapon platforms and ground support equipment. This anodize process forms a thick oxide film which provides more protection against chemical degradation than chemical conversion coatings. MIL-A-8625E "Anodic Coatings, for Aluminum and Al Alloys" Type I covers the performance requirements of chromic acid anodizing (CAA). While this chromated anodize process offers satisfactory performance, future restrictions necessitate the elimination of chromium emissions from 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). This can be accomplished by material substitution or by the use of alternative technologies, which provide the same overall properties. While both of these methods reduce the amount of hazardous material released, only the former solves disposal and handling concerns. In addition, the elimination approach to chromic acid anodizing will significantly reduce the total amount of chromium emitted from Navy operations and is in direct support of Navy and DOD hazardous waste minimization policies and directives. The need for expensive control equipment required by 1994 under current AQMD laws would be eliminated, resulting in significant cost avoidance. Control equipment for the six Navy Depots was initially estimated at $4.5-6M for capitol costs and $2.5-4M for annual operating costs. An adequate replacement would provide protection against excessive environmental degradation. This is particularly important considering the severely deleterious environment in which the Navy operates, as well as the cost of the aircraft, weapon systems and ground support equipment.

The Naval Air Warfare Center Aircraft Division at Warminster (NAVAIRWARCENACDIVWAR) has an extensive environmental materials program aimed at the elimination of hazardous materials from Navy aerospace processes (Ref 3). An evaluation effort under these programs was established at NAVAIRWARCENACDIVWAR to demonstrate an alternative technology as a replacement for CAA. The following is a description of this program.

DESCRIPTION OF SURFACE 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 chromic acid anodizing control process used in this investigation is covered by MIL-A-8625E Type I. Specific details on chromic acid anodizing of aluminum are provided in references 5-6.

Two potential alternatives have been identified for replacement of chromic acid anodizing. These alternatives are: Boeing Aerospace Corp's Boric Sulfuric Acid Anodize (SBAA) and thin film sulfuric acid anodizing (Refs 7 - 9). During the initial stage of this effort, the available test data on the two processes was analyzed. Due to the greater volume of existing SBAA data, this process was selected for demonstration.

To evaluate this process, a laboratory scale sulfuric/boric acid anodize process line was set up at NAVAIRWARCENACDIVWAR. This operation was used to analyze the performance properties of sulfuric/boric acid anodizing in comparison to chromic acid anodizing, both sealed and unsealed on various substrates. These films were examined as pretreatments for standard Navy coatings using the procedures described below. Also, the fatigue characteristics of the SBAA and CAA oxides were characterized to determine any detrimental effects. Several coating weights were examined for their effect of fatigue life degradation. A test report on the full scale fatigue tests is being prepared as a separate document, however a summary of the tests is included in Appendix A.

In addition, a 3,200 gallon production scale SBAA line was installed at the North Island Naval Aviation Depot in San Diego, CA. This facility was used to process selected test components for evaluation and optimization of the SBAA process. These results were used to determine the effectiveness of this non-chrome alternative to provide equivalent corrosion resistance and paint adhesion, while maintaining the existing mechanical properties provided by chromic acid anodizing.

EXPERIMENTAL

The performance of both the SBAA and CAA processes was 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

With the exception of those panels which were used to determine coating weights, 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 NAVAIRWARCENACDIVWAR and North Island NADEP 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). Chromic acid anodized control specimens represent a common pretreatment found on military aircraft prior to painting.

Anodize Seals

The chromic 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 acid anodize specimens were sealed with a dilute chromic acid seal described in reference 7.

Experimental Procedures

Coating Weight Determination

Coating weights of the anodize films were obtained using the test procedure outlined in MIL-A-8625E. Weights for sealed and unsealed films were determined on several different alloys. Both anodize processes were included and 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/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

TABLE 1: ORGANIC COATINGS SPECIFICATIONS

1. MIL-P-23377D, Type 1 "Primer Coatings, Epoxy Polyamide, Chemical and Solvent Resistant." Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

2. MIL-P-85582A, Type 1 "Primer Coatings: Epoxy, Waterborne." Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

3. TT-P-2760, Type 1 "Primer Coating: Polyurethane, Elastomeric." Film thickness: 20.3 to 30.5 microns (0.0008 to 0.0012 inches).

4. MIL-P-23377D, Type 1, Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).
   MIL-C-85285B, Type 1, "Coating: Polyurethane, High Solids." Film thickness: 45.7 to 55.9 microns (0.0018 to 0.0022 inches).

The above coatings were applied by conventional air spray and were 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

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, then chromic acid anodized or sulfuric/boric acid anodized. 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, tested were specimens primed with MIL-P-23377 and topcoated with MIL-C-85285 high solids polyurethane. These coatings are described in references 10-13. All materials met the corrosion resistance and coating adhesion requirements of MIL-A-8625 anodic coatings, for aluminum and aluminum alloys. The following is a summary and discussion of the results.

Coating Weights

Table 3 shows coating weights for the two anodize processes on 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 processes resulted in films within the current proposed limits (200-700 mg/ft^2) for the F revision of MIL-A-8625 specification. Some of the sealed SBAA specimens were not evaluated due to a problem (leak) with the seal tank in the demonstration process line. However, the relative coating weights for unsealed specimens indicate that these would be in line with the proposed limits.

Adhesion/Water Resistance

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 Table 4. A standard aerospace requirement for scrape adhesion is 3 kg. The scrape adhesion results for chromic acid anodize ranged from 0.5 Kg to >10.5 Kg. This indicated that other factors (such as the coating edge effects, pretreatment thickness,

TABLE 3. COATING WEIGHT RESULTS

AL ALLOY	Sulfuric/Boric Acid Anodize Coating Weight (mg/ft^2)	Chromic Acid Anodize Coating Weight (mg/ft^2)
2024-T3 (Sealed)	372.8	576.4
7075-T6 (Sealed)	576.0	567.2
6061-T6 (Sealed)	-----	366.9
7075-T6 Alclad (Sealed)	-----	436.3
3003 (Sealed)	-----	359.5
1100 (Sealed)	-----	449.6
2024-T3 (Unsealed)	150.9	416.0
7075-T6 (Unsealed)	429.9	315.5
6061-T6 (Unsealed)	568.8	370.9
7075-T6 Alclad (Unsealed)	635.5	421.1
3003 (Unsealed)	488.5	357.1
1100 (Unsealed)	584.5	428.8

TABLE 4. ADHESION/WATER RESISTANCE TEST RESULTS

ALLOY/ COATING	Sulfuric/Boric Acid Anodize SCRAPE (Kg)	WET TAPE (24*)	WET TAPE (96)	WET TAPE (168)	Chromic Acid Anodize SCRAPE (Kg)	WET TAPE (24)	WET TAPE (96)	WET TAPE (168)
2024-T3 Al Alloy								
MIL-P-23377	3.0	5A	5A	5A	2.0	5A	5A	5A
MIL-P-85582	1.5	5A	5A	5A	2.0	5A	5A	5A
TT-P-2760	1.5	5A	5A	5A	0.5	5A	5A	5A
23377/MIL-C-85285	---	5A	5A	5A	---	5A	5A	5A
7075-T6 Al Alloy								
MIL-P-23377	7.0	5A	5A	5A	10.5	5A	5A	5A
MIL-P-85582	2.0	5A	5A	5A	2.5	5A	5A	5A
TT-P-2760	1.5	5A	5A	5A	0.5	5A	5A	5A
23377/MIL-C-85285	---	5A	5A	5A	---	5A	5A	5A

--- Test not performed
* Hours immersion in deionized distilled water

Results are for panels processed at NADEP North Island, San Diego, CA

pre-paint surface cleanliness, etc.) affected the outcome of the tests. The results from the sulfuric/boric acid anodized process ranged from 1.5 Kg to 7.0 Kg and was comparable to chromic acid anodizing. The 24 hour tape tests performed on these processes showed adhesion values of 5A for all three primers evaluated. These results indicate virtually no susceptibility to coating-substrate disbondment upon exposure to water. In the expanded adhesion tests (4 & 7 days) both anodize processes, with various coating systems, continued to exhibit excellent adhesion & water resistance. This is evidenced by the tape test 5A results after extended immersion in water.

Bare Corrosion Resistance

Sealed, unpainted specimens from both 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 5. Both anodic processes on all alloy specimens passed 336 hours of exposure without any evidence of surface corrosion, indicating excellent anodic coating performance.

Painted Corrosion Resistance

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 both 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 Table 6. Both of the anodize processes, with all three primers and the epoxy primer/polyurethane topcoat coating system, passed 1000 hours of exposure. There were little to no corrosion products in the scribe and no blistering of the coatings.

Since both systems performed well for over 1000 hours on both substrates, the test was continued for another 1000 hours. At 1500 hours and 2000 hours, there were little to 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 2000 hours, all of the primed and primed/topcoated specimens passed the corrosion test requirements.

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 Table 7. The SO_2/salt spray environment simulates industrial stack gases, such as those found on diesel powered aircraft carriers, and it is an extremely aggressive environment. Most aircraft coating specifications do not have exposure to SO_2/salt spray as a corrosion

TABLE 5. 5% NACL SALT SPRAY RESULTS FOR UNPAINTED PANELS

ALLOY	-	ANODIZE PROCESS	336 HOUR TEST RESULTS
2024-T3	-	Sulfuric/Boric	No surface corrosion
2024-T3	-	Chromic	No surface corrosion
7075-T6	-	Sulfuric/Boric	No surface corrosion
7075-T6	-	Chromic	No surface corrosion

Results for panels processed at NADEP North Island

TABLE 6. CORROSION RESISTANCE TEST RESULTS (5% SALT SPRAY)

ALLOY/ COATING	SULFURIC/BORIC ACID ANODIZE HOURS OF EXPOSURE				CHROMIC ACID ANODIZE HOURS OF EXPOSURE			
	500	1000	1500	2000	500	1000	1500	2000
2024-T3 Al Alloy								
MIL-P-23377	P	P	P	P	P	P	P	P
MIL-P-85582	P	P	P	P	P	P	P	P
TT-P-2760	P	P	P	P	P	P	P	P
23377/MIL-C-85285	P	P	P	P	P	P	P	P
7075-T6 Al Alloy								
MIL-P-23377	P	P	P	P	P	P	P	P
MIL-P-85582	P	P	P	P	P	P	P	P
TT-P-2760	P	P	P	P	P	P	P	P
23377/MIL-C-85285	P	P	P	P	P	P	P	P

P = Pass (no blistering, pitting or uplifting of coating, but trace amounts of corrosion permitted in scribe)
+ = Borderline Pass (some corrosion in scribe, but no blistering or pitting on panel)
- = Borderline Failure (corrosion in scribe with initiation of blistering or pitting on panel)
F = Failure (corrosion and/or blistering in scribe and on panel surface)

Results are for panels processed at NADEP North Island, San Diego, CA

TABLE 7. CORROSION RESISTANCE TEST RESULTS (SO_2/SALT SPRAY)

ALLOY/ PRETREATMENT	SULFURIC/BORIC ACID ANODIZE HOURS OF EXPOSURE			CHROMIC ACID ANODIZE HOURS OF EXPOSURE		
	168	336	500	168	336	500
2024-T3 Al Alloy						
MIL-P-23377	P	P	F	P	P	F
MIL-P-85582	F			F		
TT-P-2760	F			F		
23377/MIL-C-85285	P	+	-	P	+	-
7075-T6 Al Alloy						
MIL-P-23377	P	+	-	P	-	-
MIL-P-85582	-	F		+	F	
TT-P-2760	F			F		
23377/MIL-C-85285	P	F		P	-	F

(P = Pass, + = Borderline Pass, - = Borderline Failure, F = Failure)

Results are for panels processed at NADEP North Island, San Diego, CA

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. All specimens (both alloys & both processes) primed with MIL-P-85582 and primed with TT-P-2760 had some scribe corrosion and slight blistering of the coating at 168 hours. These primer specimens failed, except for the MIL-P-85582 on 7075 which was a borderline result for both processes. All MIL-P-23377 primed specimens passed at 168 hours. Primed and topcoated 7075 specimens with both processes failed completely at 500 hours or less, while the primed and topcoated 2024 specimens with both processes were considered a borderline fail at 500 hours. Here again both anodize processes failed at relatively the same exposure time.

In general, the corrosion resistance of the sulfuric/boric acid anodize processed specimens 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 this non-chromated anodic coating as compared to the chromated anodic coating, is due to a high degree of interfacial integrity between the coating and substrate.

Finally, the fatigue results in Appendix A show that the SBAA process provided equivalent fatigue characteristics to the CAA process. A statistical analysis of variance performed on the results showed no significant differences in performance between the two anodize processes, although the mean value for fatigue life of the SBAA appeared to be slightly better than CAA.

SUMMARY

The goal of this project was to demonstrate a non-chromated alternative for chromic acid anodizing used on current aerospace structures. The results from this evaluation show that the two anodize processes have comparable performance properties. Having successfully demonstrated this process at the North Island Naval Aviation Depot, the full scale use as an alternative to chromic acid anodizing is being pursued. Transitioning and full implementation of this process for use in fleet maintenance operations is being accomplished through the modification of the MIL-A-8625 military specification. The use of this 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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APPENDIX A

FATIGUE TEST RESULTS*

ANODIZE PROCESS	COATING WEIGHT (mg/sq ft)	MEAN FATIGUE LIFE (Cycles)
None	N/A (Baseline)	93,000
SBAA	200	74,000
CAA	200	67,900
SBAA	500	67,500
CAA	500	64,400

* See Test Report for More Details

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