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✓ CHROMATE CONVERSION COATING OF ALUMINUM ALLOYS

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INTRODUCTION

The application of chromate coatings to aluminum often results in films with acceptable appearance and adhesion but poor performance characteristics. For some years the effect of processing variables on the integrity of chromate conversion coatings formed on high strength aluminum alloys has been under investigation. Results have been published in various reports. Due to the fact that Navy aerospace contractors from time to time experience difficulty with their production processing facilities, it appeared desirable to bring together in one document the knowledge gained during these studies leading to the establishment of what is considered to be an optimum processing procedure.

SUMMARY OF RESULTS

Chromate conversion coatings that provide maximum corrosion protection are applied after non-etch alkaline cleaning and deoxidizing with a chromate-sulfate deoxidizer. Adequate rinsing between each step is important.

CONCLUSIONS

Performance of a chromate conversion coating on aluminum alloys, particularly with regard to corrosion resistance, is heavily influenced by each step in the processing cycle—cleaning, deoxidizing, chromating, rinsing and drying.

Corrosion resistance is also influenced by the composition and heat treatment of the aluminum alloys as well as mill practices employed in their production.

RECOMMENDATIONS

The following processing cycle is recommended for use in chromating aluminum alloy components for aerospace usage:

1. Clean in non-etching alkaline cleaner.
2. Rinse at least one minute.
3. Deoxidize using chromate-sulfate deoxidizer.
4. Rinse at least one minute.
5. Apply a MIL-C-81706, Class 1A chromate film.
6. Rinse film at least one minute.
7. Air dry at temperatures below 140°F.

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B A C K G R O U N D

Chromate conversion coatings are covered by two military specifications (references (a) and (b)). MIL-C-81706 is used for the purpose of qualifying the materials themselves to certain requirements and materials meeting the requirements are placed on a qualified products list. MIL-C-5541 covers the films applied in production and is essentially a quality control specification.

There are two classes of films under MIL-C-81706. Class 1A film must pass a two-week salt spray exposure (ASTM B117) with no sign of corrosion whatsoever, and also pass several paint adhesion tests. Alloys are 2024-T3 and 7075-T6. Class 3 films have low electrical resistance and must pass a one-week salt spray test on 6061-T6 alloy with no sign of corrosion.

P R O C E S S D E S C R I P T I O N

The usual steps in conversion coating aluminum alloys are: cleaning, de-oxidizing, chromating, rinsing and drying. Within each step there are variables that can greatly influence the end product. For cleaning, both etch and non-etch cleaners are available; for deoxidizing, both chromated and non-chromate deoxidizers, etc. The section to follow will treat, in detail, the effect of processing variables in each step. A variable also present is the aluminum alloy itself that is being treated, its chemical composition, heat treatment and mill history in general.

Effects of Alkaline Etch and Non-etch Cleaning

Nine alkaline non-etch and seven alkaline etch proprietary cleaners were evaluated to determine their effect on the corrosion resistance of chromate coated 2024-T3 aluminum alloy (reference (c)). Included among the non-etch cleaners were three silicated and two non-silicated (one was chromate inhibited). Included among the etch cleaners were light, mild and heavy duty etches. New deoxidizing-desmutting and MIL-C-81706 Class 1A chromate film solutions were prepared for the study, and were again prepared new after the evaluation of eight cleaners. The film was applied by immersion.

Sixteen sets of panels were cut from the same .032-inch sheet of 2024-T3 aluminum. A set consisted of eight 3-inch by 10-inch panels. Each set was processed on the same rack through the cleaning, deoxidizing-desmutting and chromating solutions. Five panels from each set were exposed to 5 percent salt spray as per ASTM Method B117 and examined for corrosion at 168 and 336 hours. Two panels were coated with MIL-P-23377 epoxy primer and MIL-L-81532 acrylic lacquer topcoat. Knife and wet tape adhesion tests were made on the paint films. Results are presented in Tables I and II.

Salt spray exposure for 168 hours did not result in pitting corrosion on any of the nine 2024-T3 sets that were non-etch cleaned, deoxidized and chromate coated. Severe pitting corrosion was observed on four of seven alkaline cleaned sets, while two sets met MIL-C-5541B salt spray requirements. (MIL-C-5541B has the following corrosion resistance requirement: Test piecesshall show no more than a total of 15 isolated pits none larger than

T A B L E I

**NON-ETCH CLEANER PRETREATMENTS FOR CHROMATED 2024-T3
PANELS AND SALT SPRAY RESULTS**

<u>Cleaner Description</u>	<u>Deoxidizer- Desmutter Immersion Time</u>	<u>Salt Spray Exposure</u>	
		<u>No. Pits on Panels</u>	<u>168 Hours</u>
Non-etch, silicated	7 minutes	0,0,0,0,0	0,0,0,0,2
Non-etch, mildly alkaline, biodegradable surfactants	3 minutes	0,0,0,0,0	1,0,0,0,1
Non-etch, alkaline, soak cleaner	10 minutes	0,0,0,0,0	0,0,0,0,0
Non-etch, nonsilicated	6 minutes	0,0,0,0,0	0,0,0,0,0
Non-etch, nonfoaming	10 minutes	0,0,0,0,0	0,0,0,0,2
Non-etch, silicated	9 minutes	0,0,0,0,0	0,0,0,0,0
Non-etch, nonsilicated chromate inhibited	3 minutes	0,0,0,0,0	0,0,0,0,0
Non-etch, silicated	4 minutes	0,0,0,0,0	0,0,0,0,0
Non-etch, alkaline	6 minutes	0,0,0,0,0	0,0,0,0,0

T A B L E I I

ETCH CLEANER PRETREATMENTS FOR CHROMATED 2024-T3
PANELS AND SALT SPRAY RESULTS

Cleaner Description	Deoxidizer- Desmutter Immersion Time	Salt Spray Exposure	
		No. Pits on Panels 168 Hours	336 Hours
Mild etch, nonsilicated	3 minutes	0, 0, 0, 0, 0	0, 2, 0, 0, 0
Fine uniform etch	7 minutes	20, 9, 38, 28, 2	20, 10, 40, 30, 10
Light etch, biodegradable surfactants	7 minutes	18, 24, 8, 8, 7	18, 24, 11, 9, 13
Heavy duty etch	7 minutes	0, 0, 8, 0, 6	0, 9, 8, 0, 16
Heavy duty etch	6 minutes	0, 0, 0, 0, 0	1, 0, 0, 0, 0
Bright alkaline etch	5 minutes	80, 35, 12, 85, 30	85, 35, 15, 88, 35
Etch, nonsilicated biodegradable surfactants	4 minutes	35, 50, 20, 35, 40	45, 60, 30, 55, 55

1/32 in. in diameter in a total of 150 square in. of exposed test area, nor more than 5 isolated spots or pits in a total 30 in. of exposed area (also less than 1/32 in. in diameter).) One etch cleaned set was a borderline failure with less than 15 pits on five panels (150 square inches), but more than 5 pits on one panel (30 square inches). Etchants can be used for cleaning aluminum surfaces prior to the application of chromate films if (1) MIL-C-5541B quality assurance provisions are satisfied and (2) the procedure is specified in the contract or order.

The type of cleaner is not a significant variable with regard to paint adhesion on laboratory prepared panels since adhesion on all chromated panels from both non-etch and etch cleaned sets of this study was satisfactory. This observation may not apply in production, however, or where conditions are less than optimum.

Immersion times of 2024-T3 panels in the deoxidizer-desmutter solution ranged from 3 to 10 minutes. In this investigation, various deoxidizing or desmutting periods were required depending on the cleaner in order to obtain uniform surface cleanliness based on visual inspection. Surface films deposited by non-etch cleaners varied with cleaner composition, immersion time and immersion temperature. Smut removal times varied with the etch cleaners due to different etch rates and other processing variations.

The cleaning procedure is obviously one of the key variables in the preparation of a corrosion resistant finish. MIL-C-5541B requires cleaning with non-etch cleaners. Some military equipment contractors and subcontractors prefer etch cleaning since it is used in other aluminum finishing operations. Etch cleaning of aluminum results in dimensional changes that are undesirable for parts with close tolerance fits such as fastener holes. However, etch cleaning would be acceptable on parts without critical fits or strength levels provided there were no other adverse effects.

The results of this study supported the MIL-C-5541B requirement that chromate films be applied to non-etch cleaned aluminum surfaces. It should be mentioned here that non-etching alkaline cleaners will remove light soil, oils and greases. Heavy soils, oils and greases may require a degreasing step prior to alkaline cleaning.

Effect of Deoxidizing

The inclusion of a deoxidizing step in the process is highly recommended because this removes all traces of alkaline cleaning products and activates the surface for subsequent chromating. A study has been made of the effect of mechanical deoxidizing compared with several types of chemical deoxidizers again with regard to corrosion resistance and also electrical resistance (reference (d)). Chemical deoxidizers were a proprietary chromate-sulfate, two proprietary non-chromate deoxidizers and a sodium sulfate-nitric acid solution sometimes used to clean aluminum prior to spotwelding. Immersion times were varied in the chromate-sulfate solution to study this effect on corrosion resistance. One set of panels was prepared using abrasive nylon pads for mechanical surface oxide removal followed by non-etch cleaning.

For corrosion tests 2024-T3 aluminum alloy panels were prepared from .032 in. thick inventory sheet stock. Five panels from each set were exposed to 5% salt spray and a sixth panel served as an unexposed control to evaluate the corrosion test results. The same non-etch cleaner and chromate solution were used for the deoxidizer variation study. Application method for the chromate was immersion.

Panels for electrical resistance tests were 3" by 10" cut from .032 inch thick 6061-T6 aluminum alloy sheet. A set of three panels was processed at each treatment condition. Surface resistance tests were run on sets non-etch cleaned only, and non-etch cleaned followed by deoxidizing. Tests were conducted in accordance with section 4.5.5 of MIL-C-81706. Variation effects were studied using a proprietary chromate-sulfate deoxidizer, a proprietary chromate-nitrate deoxidizer, two proprietary non-chromate deoxidizers, and a sodium sulfate-nitric acid deoxidizing solution. All the deoxidizers were evaluated at two immersion times.

The salt spray results are given below in Table III for the various deoxidizers.

TABLE III

DEOXIDIZER EFFECT

<u>Deoxidizer</u>	<u>Average Number Pits</u>
Abrasive pad	1
Chromate-sulfate	3
Non-chromate #1	35
$\text{Na}_2\text{SO}_4\text{-HNO}_3$	60
Non-chromate #2	74

Mechanical removal with nylon mesh pad or chemical surface activation by the 2-minute immersion in a chromate-sulfate solution provided the most improvement in corrosion resistance. There was no definite correlation of deoxidizing immersion times with corrosion resistance, but the treatments with short immersion times yielded fewer pits after salt spray testing.

The electrical resistance test results of 6061-T6 test panel surfaces deoxidized in several different solutions are given in Table IV.

The best results with low resistances and small standard deviations were obtained on panels treated with a chromate-nitrate deoxidizer. Surfaces non-etch cleaned, but not deoxidized, gave test results of 1800 microhms per square inch with a large standard deviation of 1460. Panels deoxidized in a proprietary chromate-nitrate solution for 5 to 15 minutes gave low consistent surface resistances of 170 to 175 microhms. A proprietary chromate-sulfate deoxidizer gave surface resistance readings of 230 microhms for a 2 minute immersion and 170 microhms for a 10 minute immersion treatment. Good results were also obtained with sodium sulfate-nitric acid and an 8 minute treatment in one non-chromate proprietary solution. Average resistance readings for these treatments were 190 to 210 microhms per square inch.

TABLE IV

6061-T6 TREATMENTS AND ELECTRICAL RESISTANCE TEST RESULTS

<u>Deoxidizing System</u> ⁽¹⁾	<u>Immersion Time</u>	<u>Average Electrical Resistance</u> ⁽²⁾ <u>microhms/in² at 200 psi</u>	<u>Standard Deviation</u> <u>± σ</u>
None	----	1800	1460
Chromate-sulfate	2 min.	230	40
Chromate-sulfate	10 min.	170	5
Chromate-nitrate	5 min.	170	5
Chromate-nitrate	15 min.	175	5
Non-chromate #1	20 sec.	1320	1030
Non-chromate #1	2 min.	1360	750
Non-chromate #2	3 min.	445	110
Non-chromate #2	8 min.	200	0
Na ₂ SO ₄ -HNO ₃	2 min.	210	10
Na ₂ SO ₄ -HNO ₃	5 min.	190	10

(1) Cleaning system - Non-etch Alkaline - 10 minutes.

(2) Average for 30 readings

Longer treatment times in three of the solutions gave lower surface resistances. In the other two solutions there was a slight, but insignificant higher, average resistance. Standard deviations were lower, or unchanged, with longer treatment periods for all five deoxidizing solutions.

This study showed that the deoxidizing step is another key variable in the processing cycle. Mechanical deoxidizing with abrasive nylon mesh pads or a chromate sulfate deoxidizer produced the best, most consistent results with regard to corrosion resistance. The chromate-nitrate deoxidizer was also satisfactory for producing films of low electrical resistance.

Chromating Solution

Chemical films on aluminum alloys can be formed in phosphate, chromate-phosphate or all chromate solutions. Only films formed in all chromate solutions, however, will meet the stringent corrosion resistance requirements of MIL-C-81706, and even among the many proprietary chromate solutions available there is a wide range in quality. This is evident from the large number of chromate materials submitted for qualification which fail to meet the specification requirements.

Likewise not all the films on the present Qualified Products List are equal in performance. Some produce solutions that are more stable and easier to keep in control on a production basis than others, a parameter not considered during qualification. Both life and frequency of need for adjustment and replenishment of the chemicals can only be determined in production use. Some materials will require close control of concentration and pH, others have more latitude.

Some years ago a study was made of seventeen different chemical films. Corrosion resistance was determined by 5% salt spray exposure and outdoor exposure at a marine location in Florida for one year (reference (e)). A wide variation in corrosion protection was evidenced.

More recently three proprietary chromates were tested on 2024-T3 using the manufacturers recommended bath make-up and immersion procedures (reference (d)). Two were listed on the qualified list for MIL-C-81706; one was not. One material was tested at different concentrations and solution make-up conditions. The effects of the different chromate treatments were studied on surfaces pre-treated by non-etch cleaning and chromate-sulfate deoxidizing. Results are shown in Table V.

TABLE V

EFFECT OF CHROMATING SOLUTION ON CORROSION RESISTANCE

<u>Treatment</u>	<u>Average Number Pits*</u>
Chromate #1	3
Chromate #2	5
Chromate #3	55

EFFECT OF CONCENTRATION AND MAKE-UP WATER ON CORROSION RESISTANCE

<u>Chromate Solution #1</u>	<u>Average Number Pits*</u>
Recommended concentration/distilled water	3
2X concentration/distilled water	15
1/2X concentration/distilled water	18
Recommended concentration/tap water	16

* Average from five 3" x 10" panels

The different chromate treatment materials and solution make-up methods gave a range of test results. The most corrosion resistant films were formed in the control chromate bath when used in accordance with the manufacturer's instructions.

The chromate can be applied by three methods: brush, immersion or spray. The early study on the seventeen films indicated that brush and immersion methods produced better, more consistent results in corrosion resistance and paint adhesion than did the spray method. This could be expected since the solution

is in more intimate contact with the metal during immersion or brush treatment.

A recent laboratory development now undergoing field testing is a sprayable/brushable thixotropic chemical conversion coating specifically formulated for corrosion protection of aircraft skin surfaces and components (reference (f)). The thickened material was designed to eliminate application problems such as rapid run off from vertical and curved surfaces. The chromate is thereby in contact with the surface for a longer time and a more protective film is formed. The thickened version is in conformance with the requirements of MIL-C-81706.

The proprietary chromates also vary in the accelerator used. The most common are ferricyanide, molybdate or tungstate salts with the ferricyanide ion considered to be the most effective accelerator.

Water for Bath Make-Up and Rinsing

It is very important to know the analysis of water used for make-up of baths and for rinsing. Excessive chloride, fluoride or sulfate can result in chromate solutions that are too reactive, while phosphate ions hinder film formation from a chromate conversion solution. On the other hand, small amounts of these anions, especially chloride, are necessary to promote film formation. The water make-ups may require treatment such as going through an ion exchange column. Frequently it is necessary to use a blend of the regular water available and distilled or deionized water in making and maintaining chromate conversion baths.

High concentration of certain cations such as calcium, barium, magnesium and iron react with important bath components forming low solubility or inert materials.

Rinsing

Adequate rinsing between cleaning and deoxidizing steps is important to minimize drag-over of the alkaline cleaner into the acid deoxidizer. Rinsing between deoxidizing and chromating steps is also important to avoid carry-over of sulfate, nitrate or phosphate ions that will affect the film forming reaction as discussed in the previous section. Particular care should be taken with parts of intricate geometry where solution could be trapped.

It is important to rinse following chromating in order to remove insoluble salts which could degrade corrosion resistance. Most manufacturers of chemical film materials specify overflowing water rinses. Some recommended a final acidulated rinse. The water can be heated to facilitate drying, but not above 140°F to avoid leaching the hexavalent chromium out of the film. Spray rinses are also acceptable.

The length of time in the rinse tank is largely dependent on the degree of agitation in the tank and the geometry of the part. A minimum of one minute for each rinse is recommended.

Drying

Drying at temperatures above 140°F should be avoided. Elevated temperatures insolubilize the hexavalent chromium in the film and lower its corrosion protective properties. It is not temperature alone that causes film deterioration—there is also a time/temperature effect. Long term exposure at temperatures lower than 140°F can be just as detrimental to corrosion resistance if excessive dehydration takes place.

Effect of Alloy Composition and Heat Treatment

The lower the alloy content of the aluminum alloy the heavier will be the films formed in equivalent immersion times in the chromate solution. A study was made of the amount of hexavalent and trivalent chromium in films formed on pure aluminum, Alclad 2024 (cladding alloy is 1230 which is 99.3% minimum aluminum) and bare 2024 which contains nominally 4.4% copper and 1.5% magnesium. Processing steps were the same for all, and a three minute immersion time was used. Hexavalent chromium was leached from the films by boiling in distilled water. The remaining film was stripped in warm, concentrated nitric acid. Chromium determinations were made colorimetrically (reference (g)). Results are given in Table VI.

TABLE VI

EFFECT OF ALLOY COMPOSITION

<u>Basis Metal</u>	<u>Total Amount of Chromium</u> (micrograms/in ²)
Pure aluminum	375
Alclad 2024 (1230)	197
Bare 2024	129

These values demonstrate that the lower the number of alloying elements present, the more chromium the film contained; i.e., the heavier the film (reference (h)).

Heat treatment also plays a part in influencing film formation. Naturally aged alloys such as 2024-T3 react differently than do artificially aged alloys such as 2024-T8 and 7075-T6. The elevated temperature aging bakes on the oxides formed during solution heat treating and quenching. A good deoxidizing treatment is especially important for removing these oxides prior to chromating.

Also, mill practices used in producing wrought alloys influence the results. Impurities may be rolled into the surface which cannot be removed by chemical means. A surface free of contaminants and impurities will naturally acquire a better film.

A study to determine effect of metal source on chromating and subsequent corrosion resistance produced interesting results. The panels used represented different aluminum producers and different lots. Intergranular corrosion susceptibility was determined by a six-hour immersion in a salt-peroxide

solution (reference (1)). Results are presented in Table VII.

TABLE VII

METAL SOURCE TEST RESULTS

Metal Source	Intergranular Corrosion Test	336 Hours Salt Spray	
		Average Pits Per Panel (1)	
		Chromate-Sulfate Deoxidizing-2 minutes (2)	Abrasive Pad Deoxidizing (3)
1	Not susceptible	17	11
2	Not susceptible	1	4
4	Susceptible	1	6
5	Not susceptible	6	2
6	Slightly susceptible	10	4
7	Not susceptible	4	2

(1) Cleaner - Non-etch Alkaline - 10 minutes
Chromate-sulfate deoxidizer

(2) Average for 4 panels

(3) Average for 2 panels

There was no correlation between intergranular corrosion susceptibility and corrosion resistance but the metal source made a difference. The difference in results was less with mechanical deoxidizing than with chemical deoxidizing indicating differences were due to surface effects. Mechanical deoxidizing removes surface impurities and contaminants more effectively than chemical deoxidizing.

CONCLUSIONS

1. All steps in the chromating process—cleaning, deoxidizing, chromate application and rinsing—affect the quality and corrosion performance of the final product.
2. Metal source, heat treatment and mill history of the aluminum alloys themselves also affect the final product.
3. Optimum processing includes non-etch alkaline cleaning, deoxidizing with a chromate sulfate deoxidizer and adequate rinsing between each step.

RECOMMENDATIONS

The following processing cycle is recommended for use in chromating aluminum alloy components for aerospace usage:

1. Clean in non-etching alkaline cleaner.
2. Rinse at least one minute.
3. Deoxidize using chromate-sulfate deoxidizer.
4. Rinse at least one minute.
5. Apply a MIL-C-81706, Class 1A chromate film.
6. Rinse film at least one minute.
7. Air dry at temperatures below 140°F.

R E F E R E N C E S

- (a) Military Specification MIL-C-81706, Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys
- (b) Military Specification MIL-C-5541B, Chemical Conversion Coatings on Aluminum and Aluminum Alloys
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- (g) G. B. Cook, "Analytical Method for Determining Hexavalent and Trivalent Chromium Present in MIL-C-5541 Type Coatings", Report No. NAEC-AML-2266 of 18 Aug 1965.
- (h) S. J. Ketcham, "Effect of Hexavalent Chromium Content on Performance of Chemical Films", Report No. NAEC-AML-2065 of 17 Nov 1964.
- (i) Military Specification MIL-H-6088, Heat Treatment of Aluminum Alloys

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NAVAIRDEVCON.	49
3 for 813	1 for 30P4
2 for 30023	1 for 30P6
1 for 03	1 for 30P7
1 for 20	1 for 301
1 for 30	1 for 302
1 for 40	30 for 3022
1 for 50	1 for 303
1 for 60	1 for 304
	1 for 305