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AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC-AML-2463 DATE 27 June 1966

TESTING OF CHEMICAL FILMS FOR ESTABLISHMENT OF A REVISED QUALIFIED PRODUCTS LIST UNDER SPECIFICATION MIL-C-5541A

PROBLEM ASSIGNMENT NO. 12-85 UNDER BUREAU OF NAVAL WEAPONS WEPTASK RRMA 03 013/200 1/F020-03-01

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ABSTRACT

All chemical films for aluminum alloys qualified under Specification MIL-C-5541 have been retested for conformance with the A revision of the specification and a new Qualified Products List has been issued.

Failures are analyzed and general observations on the various tests are given in the report.

I. INTRODUCTION

Chemical films for aluminum have been greatly improved since 1948, when most of the performance requirements in Specification MIL-C-5541 were developed. Additionally, the number of films on the Qualified Products List has increased as the films were more widely used. However, the specification requirements were not stringent and, therefore, the improved performance of certain films was not identifiable in the QPL. Consequently, the specification was completely revised, upgrading the requirements so that the better films could be more readily utilized for military applications.

This problem assignment was established for the purpose of retesting all the films on the old QPL to determine conformance to the new requirements. A new QPL has been issued based on the results of the tests.

II. EXPERIMENTAL PROCEDURES

A. Specimens

Specimens were 3" x 10" x .032" panels of 7075-T6 and 2024-T3 aluminum alloys for Classes 1 and 2, and "skin quality" 6061-T6 alloy for Class 3 tests. All the panels were furnished by the chemical film supplier with the exception of the Class 3 panels which were supplied by the Aeronautical Materials Laboratory.

B. Panel Preparation

1. Methods of preparing the various panels are described in Specification MIL-C-5541A. One set of panels of each alloy was supplied with the chemical film applied by the supplier (15 panels for Class 2, 7 for Class 1) and a matching set was prepared by AML with coating material furnished by the supplier. In the case of Class 3 panels (electrical resistance), all panels were prepared by AML according to the manufacturer's directions.

2. All paint systems were applied at the Aeronautical Materials Laboratory. The two paint systems called out in the specification were: (1) the epoxy system, consisting of one coat MIL-C-8514 wash primer, one coat MIL-P-23377 epoxy primer, and two coats MIL-P-22750 epoxy topcoat; (2) the nitrocellulose acrylic system, consisting of one coat MIL-C-8514 wash primer, one coat MIL-P-7962 lacquer primer, and two coats MIL-L-19537 nitrocellulose acrylic topcoat. Two anodized panels were painted concurrently with each group of chemical film treated panels for paint controls.

C. Testing

Testing procedures are also fully described in the Specification. Briefly, they consisted of the following:

Class 1 (Intended for use unpainted)

Two alloys: 7075-T6 and 2024-T3

One test: 336 hour salt spray exposure

Class 2 (Intended for use as a paint base)

Two alloys: 7075-T6 and 2024-T3

Five tests: (1) 168 hour salt spray exposure - unpainted
(2) 500 hour salt spray exposure - painted
(3) Knife test for paint adhesion - two paint systems
(4) Tape test for paint adhesion - two paint syetems
(5) Bend test for film adhesion - two paint systems

Class 3 (For low electrical resista-

One alloy: 6061-T6

Three tests: (1) Electrical resistance before salt spray exposure ($< 5000 \mu$ ohms/in^2)
(2) 168 hour salt spray exposure
(3) Electrical resistance after salt spray exposure ($< 10,000 \mu$ ohms/in^2)

There are three grades in the specification, A - spray, B - brush, and C - immersion application. Each grade required a separate and complete set of tests.

III. RESULTS AND DISCUSSION

Most of the interest seemed to be centered around Class 2, chemical films intended as a paint base. Results are given in Table 1. Twenty-four of the 111 films tested for this class passed.

Seventy-one of the 87 failing films failed to meet the 168 hours salt spray requirement which calls for "no evidence of corrosive attack whatever," but only 8 films out of the 71 failed on the basis of this test alone. This indicates that films inferior in this respect are apt to be inferior as paint bases also. A few were badly corroded at the end of the test period but the majority showed a moderate amount of corrosion.

Unless the supplier specifically requested that Class 1 panels be treated differently than Class 2, Class 2 panels were returned to the salt spray cabinet after 168 hours and exposed for an additional 168 hours for Class 1. It was interesting to observe that failures in the panels usually took place in 168 hours or not at all during the total period of 336 hours. The additional exposure time usually only tended to increase the size and depth of the pits, rather than form many new ones. There were exceptions but this was the general trend. Several films that failed to meet some of the paint requirements for Class 2 passed the 336 hour salt spray requirement for Class 1.

It has generally been accepted that thick films have better corrosion resistance than thin ones, but poorer paint adhesion qualities. This observation was found to be incorrect in many instances, where the vendor's instructions called for longer treatment times for Class 1 panels than for Class 2. The thinner Class 2 films often displayed better corrosion resistance than the Class 1 films. There appears to be an optimum thickness for the film for optimum corrosion resistance, beyond which the film becomes loose and powdery and below which the film is too thin to offer good protection.

The overall corrosion resistance of the basis metal plus the chemical film appears to depend largely on the basic corrosion resistance of the alloy to which the film is applied. Results of the Class 3 salt spray tests on 6061-T6 (the most corrosion resistant of the three alloys) support this observation, inasmuch as only one set of Class 3 panels failed the 168 hour salt spray requirement even though Class 3 films are normally much thinner than those applied to the less corrosion resistant 7075 and 2024 alloys for Classes 1 and 2.

Only 15 Class 2 films failed on the basis of paint tests alone and of these, 9 failed only the bend test, a test of little practical value.

The bend test specifies there shall be no separation of the chemical film from the basis metal when the painted panel is bent 180° over a 0.128" diameter mandrel. Since it is often difficult to tell whether the paint has separated from the chemical film or the film has separated from the metal, the bent panel from which the paint has spalled is exposed to salt spray for 100 hours and examined for evidence of corrosion. Corrosion of the panel is taken as evidence that the film came off with the paint during bending. This test is considered unduly harsh because the system is subjected to stresses it will never see in service. Also the bent portion is thus stressed prior to corrosion testing which alone renders the alloys more susceptible to corrosion. Several otherwise very good films have been kept off the QPL because of this one test.

Another test that has proved to be of no value is the 500 hour salt spray test for undercutting of paint. None of the films tested, not even the poorest ones, showed signs of failure at the end of the 500 hour exposure time. This is probably because all the films contain at least a small amount of leachable chromium compounds which inhibit corrosion in the scribed areas.

Although two paint systems are used for the various tests, both have to be applied over wash primer, as the specification is presently written. Adhesion characteristics depend more on the primer system than on the topcoats. It, therefore, would be more useful to eliminate the wash primer from under the epoxy system. This would also be more in keeping with current practice of the various services. The Navy is largely eliminating wash primer under the epoxy primer.

Results of the various paint tests show that the epoxy system failed the knife and tape tests more often than the nitrocellulose acrylic system while the acrylic system failed the bend test more often. Differences in the paint failures probably had more to do with topcoat characteristics than adhesion of the primer to the film since wash primer was used under both systems.

Differences in test results for the Aeronautical Materials Laboratory and supplier treated panels were encountered during the testing program. It was, therefore, decided that the treatment would be considered failing, if the suppliers' panels failed any of the tests. The theory behind this was that if the supplier could not produce a good coating with his own product, it was unlikely that anyone else could on a production basis. In the few instances when the panels treated by AML failed, and the supplier's did not, another set was prepared by AML and the tests were conducted again. If the re-runs also failed, the treatment was considered failing. This procedure resulted in only four films failing on the basis of the AML treated panels alone.

Color matching of the AML panels with the suppliers was found to be the best way to obtain similar performance results. This took some adjustment of the treatment times recommended by the supplier, and it was found that the treatment time varied with the purity of the make-up water. Treatment baths prepared with distilled water, as AML's were, generally required longer treatment times than baths prepared with tap water. Differences in the tap water in different areas would probably have the same effect.

Table 2 affords an interesting comparison of the performance of panels treated by the supplier with those treated by the qualifying activity. In the paint adhesion tests, the number of failures was greater for panels treated by the supplier by approximately 2 to 1. This could be a coincidence or it could be significant. If it is significant, the reason for this that comes most quickly to mind is the possible effect of the age of the film when it is painted. The time between treatment and painting was obviously shorter for panels treated by the qualifying activity than for those treated by the supplier. If age of the film does play a role in paint adhesion, it apparently does not play a significant role in unpainted corrosion resistance, since the number of failures in the salt spray tests were almost equal for 7075, and slightly higher for the 2024 panels treated by the qualifying activity. Likewise, the number of bend test failures were approximately equal, which gives added support to the laboratory contention that the bend test cannot be considered a test for paint adhesion.

The question of the effect of the age of the chemical film on paint adhesion has come under considerable scrutiny of late. For a while, it seemed that perhaps for qualification testing it might be fairer to standardize on a time between treating and painting. This would only be possible however if the qualifying activity treated all the panels for painting which would negate the original purpose of having comparative sets.

Analysis of elapsed time intervals on films that met all the requirements of the new specification showed that most of them had been stored for periods equally long or longer before painting than had the films that failed. In a study under Problem Assignment No. 12-79, of the effect of age and/or contamination of chromated surfaces on subsequent paint adhesion, results clearly indicated that if a good film was applied in the first place, paint adhesion was still satisfactory regardless of the age of the film. Upon further reflection then it was decided that this downgrading of paint adhesion with increasing age of the film was a characteristic of inferior or borderline films, and any procedure which eliminated such films from the QPL should be retained.

A comparison of failures of treated 2024 alloy versus treated 7075 (Table 2) shows that the total number was roughly equal for each alloy both in salt spray and in paint tests. This does not bear out the generally held opinion that it is harder to produce a good chemical conversion coating on 7075 than on 2024 because of the nature of the former's heat treat oxide.

Results of Class 1 and Class 3 tests were fairly straight forward, and there appears to be little worthy of discussion in these results.

The final outcome of the problem assignment is given in Table 3. A total of 103 treatments were submitted for Class 1 out of which 26 passed. One hundred and eleven were submitted for Class 2, 24 passed. Sixty-eight were submitted for Class 3, 13 passed.

IV. CONCLUSIONS

1. The new specification requirements are not impossible to meet, as predicted by many of the suppliers, and are adequate to screen out films of inferior or erratic performance whose use had created so many problems in the field.

2. Two tests are of little or no value:

   a. The bend test for chemical film adhesion.
   b. The 500-hour salt spray test on painted panels whose only result is unduly prolonging the testing time by tieing up salt spray facilities.

3. The use of wash primer under both paint systems does not permit the maximum amount of useful testing with the available panels and does not reflect current practice.

V. RECOMMENDATIONS

It is recommended that Specification MIL-C-5541A be amended as follows:

*1. Eliminate the 500 hour salt spray requirement for painted panels, paragraph 3.7.2.

2. Eliminate the bend test for film adhesion, paragraph 3.9.

*3. Eliminate the MIL-C-8514 wash primer from the epoxy system of paragraph 4.3.1.3.4.2 (Use MIL-P-23377 as the first coat).

*Note: Pending formal changes in the specification as recommended, these two requirements will be waived in all future retests.

CLASS 2 FAILURES LISTED BY CODE LETTERS

FILM	FAILURE							
	Unpainted Corrosion Resistance	Painted Corrosion Resistance	Knife Test		Tape Test		Bend Test	
			Epoxy	Acrylic	Epoxy	Acrylic	Epoxy	Acrylic
A	F							
B	F				F			
C-1	F			F				
C-2	F		F	F				
C-3	F		F	F				
D	F		F					
E-1	F			F		F		
E-2	F					F	F	F
F	F		F	F	F	F		
G	F							
H-1	F		F	F	F	F		
H-2	F		F	F	F	F		
I	F				F			
J	F		F	F				
K-1	F		F		F		F	
K-2	F		F			F		
L-1	F							F
L-2	F						F	F
L-3	F						F	F
M-1	F						F	F
M-2	F						F	F
M-3	F							F

F - Failure

TABLE 1

FILM	FAILURE							
	Unpainted Corrosion Resistance	Painted Corrosion Resistance	Knife Test		Tape Test		Bend Test	
			Epoxy	Acrylic	Epoxy	Acrylic	Epoxy	Acrylic
M-4	F							F
M-5	F		F		F		F	F
M-6	F							
M-7	F							F
M-8	F		F		F		F	F
M-9	F		F		F			F
M-10	F		F	F	F		F	F
N	F				F			
O	F				F			
P-1	F		F	F	F	F		
P-2	F		F		F			
P-3			F		F			
P-4	F		F		F			
Q-1	F							
Q-2	F						F	F
Q-3	F							F
Q-4	F							F
Q-5	F						F	F
Q-6								F
Q-7							F	F
Q-8								F
Q-9								F
R-1	F		F				F	F
R-2			F	F			F	F
S	F		F	F				
T-1			F	F	F	F	F	F

F - Failure

TABLE 1

F - Failure

FILM	FAILURE							
	Unpainted Corrosion Resistance	Painted Corrosion Resistance	Knife Test		Tape Test		Bend Test	
			Epoxy	Acrylic	Epoxy	Acrylic	Epoxy	Acrylic
T-2	F		F		F	F	F	F
T-3	F		F	F	F	F	F	F
T-4	F		F			F	F	F
T-5	F		F				F	F
T-6	F						F	F
T-7	F						F	F
T-8	F						F	F
T-9	F						F	F
T-10	F						F	F
T-11	F						F	F
T-12	F						F	F
T-13	F		F				F	F
T-14	F		F		F		F	F
T-15							F	F
U-1	F		F		F			F
U-2	F				F			F
U-3	F			F		F		
V	F							
W	F				F			
X-1	F			F				F
X-2	F							F
X-3	F		F	F		F		F
X-4	F			F		F	F	F
X-5	F							F
Y-1					F	F		
Y-2	F							

F - Failure

TABLE 1

FILM	FAILURE							
	Unpainted Corrosion Resistance	Painted Corrosion Resistance	Knife Test		Tape Test		Bend Test	
			Epoxy	Acrylic	Epoxy	Acrylic	Epoxy	Acrylic
Y-3	F							
Y-4								F
Y-5								F
Y-6	F							
Y-7	F							F
Y-8	F							
Z-1				F	F			
Z-2	F							
Z-3							F	F
Z-4	F						F	F
Z-5					F			F
Z-6							F	F
Z-7	F		F		F			
TOTAL	71	0	30	19	27	15	33	52
Failed on this alone	8	0	0		1		9	

F - Failure

TABLE 1

COMPARISON OF FAILURES

CLASS 2

Panels Treated by	Total Failures: Salt Spray 7075	Salt Spray 2024	Knife Test 7075	Knife Test 2024	Tape Test 7075	Tape Test 2024	Bend Test 7075	Bend Test 2024
Supplier	43	37	26	23	24	19	36	34
Qualifying Activity	46	49	12	10	13	13	38	37
TOTAL	89	86	38	33	37	32	74	71

TABLE 2

RESULTS OF REQUALIFICATION TESTS FOR MIL-C-5541A

Class	Grade*	No. Submitted	No. Passed	No. Failed
1	A	35	10	25
1	B	14	1	13
1	C	54	15	39
2	A	38	10	28
2	B	15	4	11
2	C	58	10	48
3	A	25	4	21
3	B	9	1	8
3	C	34	8	26

*A = Spray

B = Brush

C = Immersion

TABLE 3

APPENDIX A

SUGGESTIONS FOR USE OF THE NEW QPL UNDER MILITARY CONTRACTS

The characterizing of chemical films by type, grade and class may result in some confusion initially unless personnel responsible for deciding what is required for a specific application understand the significance or importance of the terms.

As a guide, the following order of precedence is suggested for making the final decision:

(1) First decide which class is required, based on the end use of the parts - that is whether they will be painted (Class 2), left unpainted (Class 1 or 2), or require low electrical resistance (Class 3). If they are to be left unpainted, the severity of the environment which will be encountered may influence the decision between specifying a Class 1 or 2 film.

(2) The second most important consideration is the grade which indicates the method of application - spray, brush or immersion. Very few contractors would have both a spray facility and immersion tanks and a decision will naturally have to be influenced by the type of facility available. For parts which are not complex, any one of three methods would be satisfactory. For parts containing recessed areas, holes, etc. which might not be reached by spraying or brushing, it would be desirable to specify the immersion method.

(3) Lastly, the selection of type is purely academic since these materials are always applied in a liquid form regardless of the method - spray, brush or dip. This decision then could be left to the discretion or convenience of the contractor. Some contractors might find storage of containers containing a powder more convenient than those containing a liquid or vice versa. Likewise, some may prefer adding water to a liquid concentrate rather than to a powder. In other words, in most cases, the choice of type will have little or no influence on the end product, but will influence the ease with which the contractor can do the work - it might even influence the cost to the Department of Defense.

UNCLASSIFIED

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DOCUMENT CONTROL DATA - R&D

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1. ORIGINATING ACTIVITY (Corporate author) NAVAL AIR ENGINEERING CENTER AERONAUTICAL MATERIALS LABORATORY PHILADELPHIA, PA. 19112	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP None

3. REPORT TITLE

TESTING OF CHEMICAL FILMS FOR ESTABLISHMENT OF A REVISED QUALIFIED PRODUCTS LIST UNDER SPECIFICATION MIL-C-5541A

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Report on Requalification Testing under MIL-C-5541A

5. AUTHOR(S) (Last name, first name, initial)

JANKOWSKY, EDWARD J.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
27 June 1966	20	NONE

8a. CONTRACT OR GRANT NO. b. PROJECT NO. Problem Assignment No. 12-85 under c. WEPTASK RRMA 03 013/200 1/F020-03-01 d.	9a. ORIGINATOR'S REPORT NUMBER(S) REPORT NO. NAEC-AML-2463 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) None

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Naval Air Systems Command Department of the Navy Washington, D. C. 20360

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DD FORM 1 JAN 64 1473

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14. KEY WORDS	LINK A		LINK B		LINK C	
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Chemical films						
Qualification testing						
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