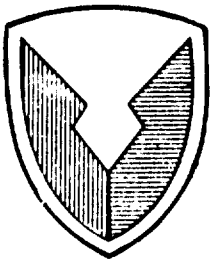

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CCL REPORT NO. 230

FINAL REPORT

A STUDY OF THE OPERATING LIMITS OF THE STANNATE IMMERSION BATH

BY

WILLIAM H. DEAVER

MAY 1967

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U. S. ARMY COATING & CHEMICAL LABORATORY

Aberdeen Proving Ground

Maryland

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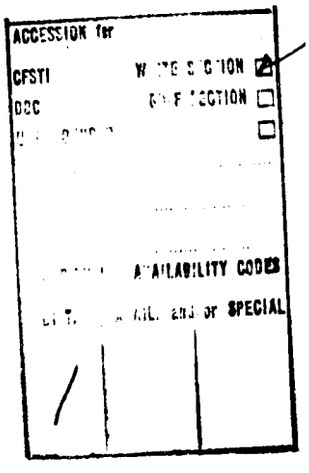

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BY

WILLIAM H. DEAVER

MAY 1967

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U. S. ARMY COATING AND CHEMICAL LABORATORY
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I. INTRODUCTION

In recent years the increased emphasis on the development of lightweight airborne equipment and missiles has caused increased use of the non-ferrous, light weight metals. Of these light weight metals magnesium has many physical properties such as ductility and a high strength to weight ratio that make it very desirable for use by the Army. Previous work (CCL Report No. 134) has shown that magnesium, with the proper pretreatment and finishing system, can be protected. However, due to design requirements it is sometimes necessary to have magnesium in contact with a dissimilar metal thereby setting up a condition for bimetallic or galvanic corrosion.

Work reported in CCL Report No. 150 showed that galvanic corrosion can be prevented by complete insulation of the dissimilar metals. It also showed that the best protection available for items containing magnesium and steel that must be treated after assembly is the stannate process developed by the Dow Chemical Company under Army contract. This is an immersion treatment which is said to deposit a tin coating on steel and a tin-magnesium salt on magnesium.

However, there has been a hesitancy to recommend the stannate process for actual production because very limited infomation was available on life of the bath, a fast effective means for replenishment of bath and maintenance of coating quality. Although an analytical method for determining bath composition was developed the procedure was much too time consuming for production control and would also require a trained analyst.

A study was therefore initiated to obtain data on the operating limits of the bath to determine if it would be possible to establish some means other than chemical analysis that could be utilized as a fast effective method for control of the process.

II. DETAILS OF TEST

To determine bath life test specimens consisting of 3 by 6 by 1/4 inch magnesium panels of alloy AZ 31 with two 1/4 inch diameter flathead steel bolts countersunk in the center of the panel 1-1/2 inches apart were stannate treated according to table I. After every 10 square feet per gallon treated, a set of four test specimens were painted to a 1 mil dry film thickness with a control formulation of MIL-P-52192, "Primer Coating, Epoxy". They were air dried 7 days, scored across one of the coupled areas and exposed to 20% salt spray in accordance with method 6061 of Federal Test Method Standard 141, along with a set treated in a freshly prepared bath.

Ninety-six hours exposure was set as the standard acceptable exposure period based on previous work that showed this was the maximum period of salt spray exposure that could be expected from stannate coatings deposited from a fresh bath without excessive amounts of galvanic corrosion. Thus a bath was considered to be depleted when the test specimens from the operating or used bath showed a faster rate of corrosion when tested simultaneously with specimens from the freshly prepared bath. Corrosion on the former generally started 24 to 48 hours sooner than those from the latter. Examples of both types are shown in photo #1.

Four separate baths were run to depletion and the amount of work treated per gallon, until substandard panels were produced, varied from 125 to 150 square feet. When the salt spray data indicated that a good coating was no longer being produced chemical analysis of the bath was made. A typical analysis is given in Table III. The potassium stannate was depleted at a much faster rate than the tetrasodium pyrophosphate. Sodium acetate and sodium hydroxide remained constant as did the pH.

A depleted bath was brought back to original strength by addition of potassium stannate and tetrasodium pyrophosphate and test panels again treated, painted and subjected to salt spray. The revived bath initially produced very satisfactory treatments. However, only 50 square feet of acceptable work was produced per gallon of bath. On close examination of bath analysis it was seen that the only constituent in the depleted bath that was not in the new bath was carbonate. It is well known that electrolytic tin plating baths of the potassium stannate type absorb carbon dioxide thereby causing poor coating formulation[2]. In order to see if the stannate immersion bath was affected in a similar manner 0.5% of sodium carbonate was added to a freshly prepared bath. Test panels were treated, painted with MIL-P-52192 and exposed to salt spray. Exposure results were very poor compared to control panels prepared in a non-carbonate stannate bath.

Since depletion rate was determined using specimens whose surface area contained 94% magnesium and 6% steel, additional studies were conducted to determine if a larger percentage of steel would deplete the bath faster. Panels were prepared with the amount of steel varying from 6% to 50% of the couple. Those containing 50% steel failed to have a coating produced on the steel. Salt spray tests (table II) showed that a magnesium-steel couple containing more than 22% steel would not receive a satisfactory stannate coating. A fresh stannate bath was run to depletion using test specimens consisting of 22% steel. It was found that 125 sq. feet could be treated per gallon before substandard coatings were produced. This indicates that the amount of steel (up to 22%) present in the couple does not affect the life or depletion rate of the stannate bath.

III. CONCLUSIONS

This study has indicated that 100 square feet of work, containing no more than 22% steel, can be safely processed per gallon of bath. Pending the development of a fast effective means for determining quality of a stannate coating, the process could be used for production with this type of control. Although it is possible to replenish the bath, it is not considered economically desirable since the life of the replenished bath is less than half that of a freshly prepared one.

IV. REFERENCES

1. Magnesium Finishing. The Dow Chemical Company, Midland, Michigan, 1963 pp. 75 - 81.

2. Blum, William and Hogaboom, George B., Principles Of Electroplating and Electroforming, McGraw-Hill Book Company, Inc., New York, 1949. p. 328.

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APPENDIX A

TABLE I

PROCEDURE FOR PREPARING STANDARD STANNATE IMMERSION PANELS

STEP 1 - Alkaline clean at 190°-212°F for 10 minutes in the following bath.

Sodium hydroxide - 120 gms
Trisodium phosphate - 20 gms
Nacconal - 2 gms
Water (to make) - 2000 ml

STEP 2 - Water rinse at room temperature.

STEP 3 - Immerse in 5% nitric acid at room temperature for 1-1/2 minutes.

STEP 4 - Water rinse at room temperature.

STEP 5 - Immerse in the following stannate bath for 20 minutes at 180°-190°F.

Potassium stannate - 100 gms
Tetrasodium pyrophosphate - 100 gms
Sodium hydroxide - 20 gms
Sodium acetate - 20 gms
Water (to make) - 2000 ml

STEP 6 - Water rinse at room temperature.

STEP 7 - Immerse in a 5% sodium acid fluoride bath at room temperature for 2 minutes.

STEP 8 - Water rinse at room temperature and dry in a 150°F oven till dry.

TABLE II

PRODUCTION OF COATINGS ON MAGNESIUM-STEEL COUPLES WITH VARYING AMOUNTS OF STEEL

Panel	Surface Area % Magnesium	% Steel	Mg:Steel Ratio	96 hours salt spray exposure
1	94	6	15.7:1	(Standard) Trace of galvanic corrosion
2	88	12	7.3:1	Equal to standard
3	78	22	3.5:1	Equal to standard
4	69	31	2.2:1	More galvanic corrosion than standard.
5	62	38	1.6:1	More galvanic corrosion than standard.
6	56	44	1.3:1	More galvanic corrosion than standard.
7	50	50	1:1	More galvanic corrosion than standard.

TABLE III

Typical Bath Composition After Depletion Of Bath By More Than 100 sq. ft. Per Gallon Of Work Treated In Bath

	Before	After
K_2SnO_3	5%	0.7%
$Na_4P_2O_7$	5%	3.5%
NaC_2H_3O	1%	1.0%
NaOH	1%	1.0%
pH	13.0	13.0
Na_2CO_3	None	Present

APPENDIX B

96 HOUR SALT SPRAY EXPOSURE

STANNATE IMMERSION

FRESH BATH

STANNATE IMMERSION

DEPLETED BATH

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13 ABSTRACT

A study was conducted to determine the operating limits of the stannate immersion process for minimizing galvanic corrosion of magnesium-steel couples. Salt spray tests on specimens treated and then painted indicated that 100 sq. feet of work containing up to 22 percent steel could be safely processed per gallon of bath before substandard coatings were produced. Magnesium steel couples containing more than 22 percent steel would not receive a satisfactory stannate coating.

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