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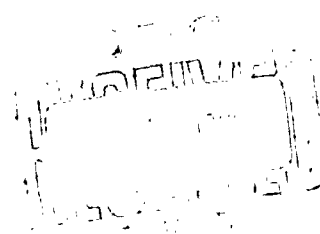
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## Rock Island Arsenal Laboratory



### TECHNICAL REPORT

MULTIMETAL COATING PROCESS FOR COMPOSITE  
STEEL, MAGNESIUM, AND ALUMINUM STRUCTURES

By

Jodie Doss

Department of the Army Project No. 1-H-0-24401-A-110

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Rock Island, Illinois

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### ABSTRACT

Aluminum, steel and magnesium trimetal assemblies were simultaneously coated in order to decrease galvanic corrosion and to develop coatings to be used for recoating structurally united components containing dissimilar metals. The following experimental solutions were utilized to coat trimetal assemblies: stannate-chromate, phosphate, stannate-gluconate, stannate-hypophosphate, and stannous pyrophosphate. The stannous pyrophosphate solution produced the best coatings on the assemblies.

#### RECOMMENDATIONS

The developed coatings from the stannate-chromate solution and the stannous pyrophosphate solution should be further investigated for use on Army materiel.

Other coatings for multimetal assemblies should be developed and investigated.

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STEEL, MAGNESIUM, AND ALUMINUM STRUCTURES**

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## MULTIMETAL COATING PROCESS FOR COMPOSITE STEEL, MAGNESIUM, AND ALUMINUM STRUCTURES

### OBJECT

To find a coating for composite steel, magnesium and aluminum.

### INTRODUCTION

Since weight is a very important consideration in the design of rockets and missiles, light metals such as aluminum and magnesium are utilized in their construction. Certain components still must be fabricated of steel where construction requirements dictate this metal.

The three metals structurally united into rockets or missiles components create two problems of major importance. The two problems are:

1. Galvanic cells - leading to galvanic corrosion (See Figure 1a).
2. Coating, to prevent galvanic corrosion without destroying one or more of the dissimilar metals in the assembly.

McKay and Worthington<sup>(1)</sup> define galvanic corrosion as the corrosion of a metal due to it being electrically connected to another metal in a corrosive condition. Such a cell is composed of four parts. The four parts are the anode, cathode, electron conductor and continuous liquid path.<sup>(2)</sup> Corrosion occurs at the anode. If one of the four parts of a galvanic cell is missing, galvanic corrosion will not occur.

Reichhard<sup>(3)</sup> states that metals are either anodic or cathodic, with respect to other metals with which they are associated. No metal has an absolute or inherent value of potential. Potential is a relative property. The potential of a metal is determined by the environment and the specific metal or metals used as a basis of comparison.

The greater the potential difference between metals, the greater the galvanic corrosion. When the potential between metals is small, the electrolyte employed determines which metal becomes the anode.<sup>(4)</sup> Coupled magnesium and aluminum exhibits a small potential, therefore, the galvanic corrosion is not great. When magnesium or aluminum is coupled with steel, the potential is relatively great.

Therefore, when steel is united with one or both of these metals in the presence of moisture, galvanic corrosion occurs.

Galvanic corrosion can be reduced in many ways. These include:

- a. Decreasing the potential by combining metals that are close in nobility, such as magnesium and aluminum.
- b. Using cathodic fasteners which result in large anodic area, in relation to cathodic area.
- c. Insulation of dissimilar metals.
- d. Polarization (passivation) of one of the metals.
- e. Painting both metals.
- f. Using corrosion inhibitors such as chromates in aqueous solution.<sup>(5)</sup>
- g. Using sacrificial anodes.
- h. Using metallic coatings to reduce contact potential of dissimilar metals.<sup>(6)</sup>

The coating of multimetal assemblies containing aluminum, magnesium and steel components to decrease galvanic corrosion creates problems. It is frequently impossible to separate the multimetal assemblies without destroying the multimetal component. It is therefore, necessary to provide a means of coating all three metals in the component simultaneously.

In coating the trimetal assemblies, one must be cognizant of the fact that alkaline solutions for coating would attack and destroy the aluminum, while acid solutions for coating would attack and destroy the magnesium. Several approaches to the problem, based upon formation of metallic coatings, appeared to offer promise of success.

It was found, during investigations of various metallic coatings for compatibility with magnesium, that tin was superior to all other metals. Some tin alloys were also good. The tin compounds were superior to cadmium and zinc on steel. Tin coatings over aluminum alloys also reduced trimetallic couple (galvanic) corrosion.<sup>(7)</sup>

Investigations were initiated utilizing solutions for tin coatings alone or coatings containing tin plus other compounds.

## PROCEDURE AND RESULTS

### Materials

The aluminum materials used in the investigation were listed under Federal Specification QQ-A-561, 1100(2S) and QQ-A-318, 5052(52S). The magnesium alloy was QQ-M-44, AZ31B.

Panels were prepared from the sheet metals in the following size, 2" x 3" x 0.0625" with 1/4" holes drilled through the center. The two aluminum and magnesium panels were securely fastened together with the major dimensions at right angle to each other, by means of a 1/4" round head steel machine screw and nut, as shown in Figure 1.

The composite specimens were degreased, utilizing a trichloroethylene vapor degreaser, prior to processing.

The following processing solutions were prepared and subsequently evaluated.

### Stannate Solutions:

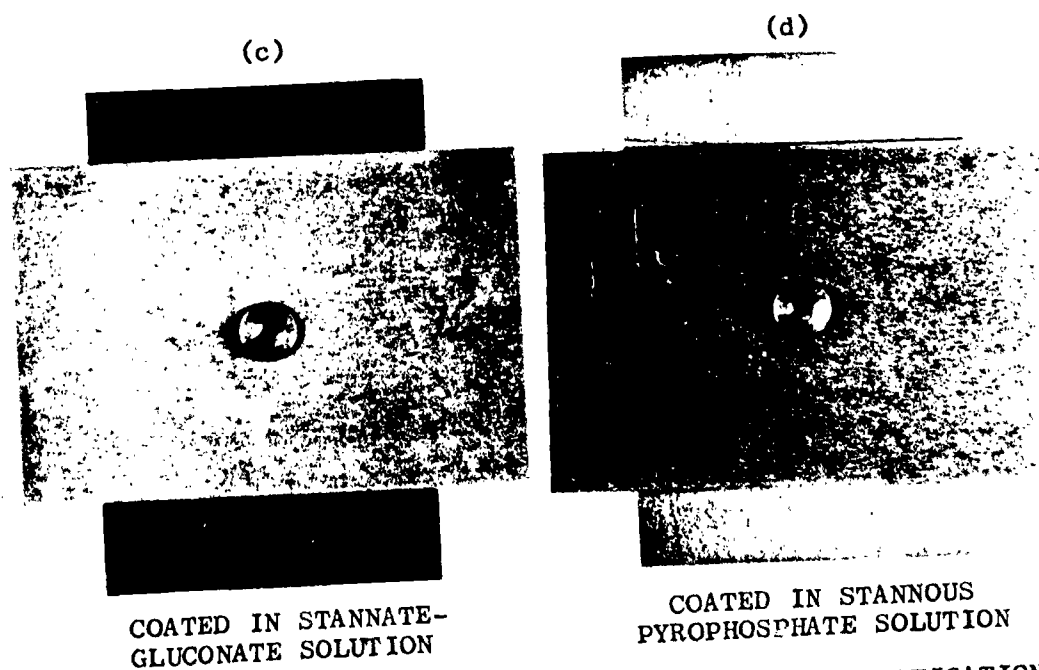
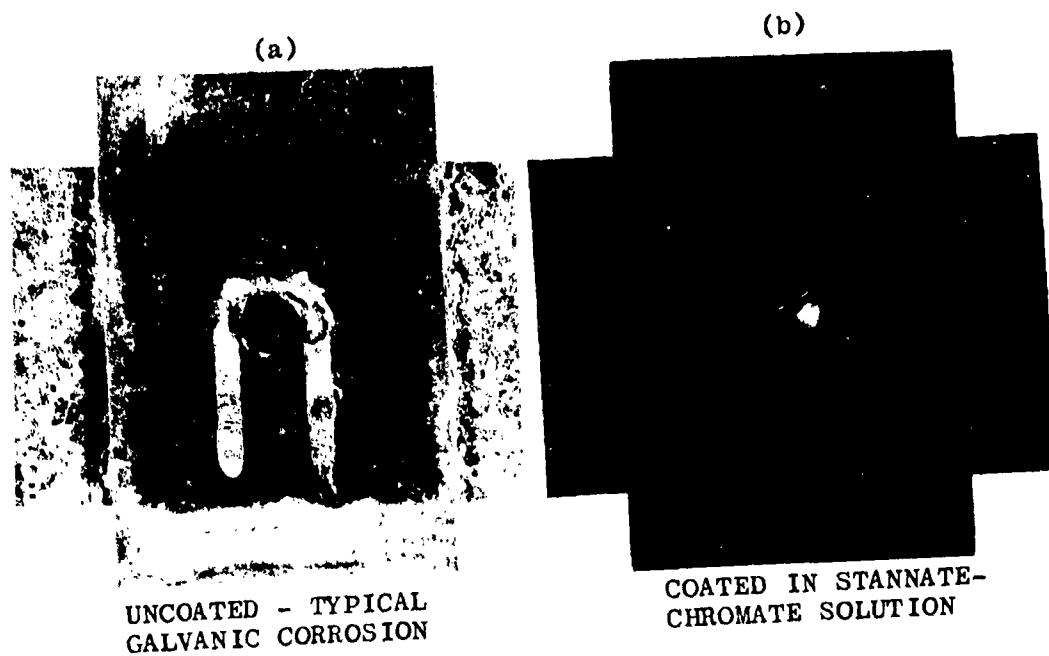
#### Chromate

A solution was prepared as shown below:

20 grams  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$   
1 gram  $\text{K}_2\text{Cr}_2\text{O}_7$   
50 grams  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$   
50 grams  $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$   
Enough water to make 1 liter of solution.

This solution was heated to  $195 \pm 5^\circ\text{F}$ . A composite trimetal assembly was submerged in the solution for thirty minutes. Upon removing the composite specimen from the solution, it was observed that a conversion coating had formed on the surface of the aluminum. Tin coatings appeared to be on the surface of the magnesium and steel (see Figure 1b). The same results were obtained with a second trimetal assembly. The addition of ten grams of sodium acetate to the solution improved the subsequent coatings. A trimetal assembly was coated in the chromate-stannate solution and submerged for ten minutes in a near boiling, 5% potassium, dichromate sealing solution, water rinsed and air blown dry. The coatings had a yellow, tinted color.

Two coated trimetal assemblies were separated and exposed, along with uncoated separated trimetal assemblies, in a 5% salt fog cabinet for corrosion resistance tests. The



TYPE OF TRIMETAL ASSEMBLIES USED IN THIS INVESTIGATION  
Vertical Panels are Aluminum, Horizontal Panels are Magnesium  
Bolts and Nuts are Steel

FIGURE 1

salt fog cabinet was operated in accordance with Method 811.1 of Federal Test Method Standard No. 151.

The test specimens were inspected after 2.5 hours. The uncoated steel had rusted. The uncoated magnesium specimen had a large amount of corrosion products on the surface. The apparently tin coated magnesium specimens had some corrosion present.

After 19 hours exposure the apparently tin coated steel screw and nut had a small amount of rust present. All the magnesium specimens (coated and uncoated) were very corroded. They were removed from further testing.

After 192 hours, the aluminum panels, believed to be coated with a chromate coating, had a small amount of white corrosion products present. The uncoated aluminum panels were dark with white corrosion products. The salt fog test was terminated.

It was found that the stannate-chromate solution would coat bimetal assemblies of steel and aluminum, steel and magnesium, aluminum and magnesium, but not steel alone. It was believed that a chromate coating was present on the aluminum and a tin coating was formed on the steel and magnesium. These coating were noted after treatment in the solution, and verified by comparison with the untreated panels after exposure in the salt fog cabinet.

It was learned that the prepared stannate-chromate solution would coat the trimetal assemblies, however, the coating on the aluminum 1100 alloy was not entirely satisfactory. It was subsequently determined, through further work, that the following solution would satisfactorily coat the alloys in the trimetal assembly.

20 grams  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$

2 grams  $\text{K}_2\text{Cr}_2\text{O}_7$

20 grams  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$

50 grams  $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$

Enough water to make a one liter solution.

The above solution was operated at  $195 \pm 5^\circ\text{F}$ .

Stannate solutions containing compounds other than chromates were prepared and attempts were made to coat the trimetal assemblies.

#### Pyrophosphate-Fluoride

The first solution contained sodium pyrophosphate and

potassium fluoride. Trimetal assemblies were processed in this solution at 180 to 190°F for twenty minutes. The pH of the solution was adjusted between 9.6 - 11.0. No satisfactory coating was obtained on any of the metals in the trimetal assemblies. The coatings were either very thin or nonadherent.

#### Ethylenediamine Tetraacetate, disodium

The second solution contained the disodium salt of ethylenediamine tetraacetic acid. The pH of the solution was adjusted to 10.3 with the acid salt. The solution was operated at 180 to 190°F. No satisfactory coating was obtained on any of the metals in the trimetal assembly. The coatings were either very thin or nonadherent.

#### Gluconate

The third solution contained sodium gluconate. The solution pH was reduced from 10.3 to 9.6 with ethylenediamine tetraacetic acid. The initial temperatures of the solution was 180 to 190°F, and then reduced to the final temperature of 140 to 150°F. Satisfactory coatings were obtained when the pH of the solution was 9.6 and the temperature of the solution was 140 to 150°F. There was an apparent bright tin coating on the steel and aluminum and a dull tin coating on the magnesium (see Figure 1c.)

The solution composition was as follows:

50 grams sodium stannate

50 grams sodium gluconate

Water to make 1 liter

This solution also coated the individual aluminum and magnesium specimens. The addition of 7 grams, per liter, of potassium fluoride to the solution improved the quality of coating on the magnesium in the trimetal assembly.

#### Hypophosphite

The fourth solution contained sodium hypophosphite. The solution was prepared as follows:

30 grams sodium stannate  $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$

10 grams sodium hypophosphite  $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$

Water to make 1 liter

The pH of the solution was adjusted to 9.5 with ethylenediamine tetraacetic acid. The solution was maintained at room temperature. Processing time was twenty minutes.

A heavy, bright, apparent tin coating was deposited on all three of the metals in the trimetal assembly. The solution coated steel plus aluminum, steel plus magnesium, magnesium and aluminum individually, but not steel alone. Two trimetal assemblies were tested in the salt spray (5%). There was very little galvanic corrosion after an 8 hour exposure.

Other compounds utilized as pH depressors, instead of ethylenediamine tetraacetic acid, were gluconic acid, boric acid and glycolic acid. The utilization of glycolic acid in the stannate solution produced a satisfactory coating on the trimetal assembly. The coating was thick and adhered to the basis metal.

The stannate-hypophosphite solution contained a large amount of sludge. It was also necessary to add, after each processing of trimetal assembly, a small amount of ethylenediamine tetraacetic acid to control the pH of the solution.

It was found that the following solutions performed satisfactorily without close process control:

10 grams sodium stannate

5 grams sodium hypophosphite

Water to make 1 liter

Initial pH of solution 11.2

The pH of the above solution was adjusted to 9.5 with 5 to 6 grams of ethylenediamine tetraacetic acid and 1 to 2 grams of boric acid. This solution was relatively free of sludge and the pH remained rather constant. The addition of one gram of  $\text{Al}(\text{NO}_3)_3$  to the solution improved the coating on the aluminum.

Trimetal assemblies previously used were cleaned to remove the deposited coatings. They were immersed in an alkaline solution containing 22 gms. sodium carbonate, 22 gms. trisodium phosphate/liter solution at 190°F for two minutes, water rinsed and rinsed in an 8%  $\text{HNO}_3$  solution. They were immediately water rinsed and placed in the stannate solution for processing.

Difficulty was encountered with rusting of the steel portion of the component after the acid dip and prior to the coating operation.

This problem was partially overcome by the addition of a small amount (.01% solution) of  $\text{I}_2\text{-KI}$  to the nitric acid

solution. The stannate-hypophosphite solution was not coating the cleaned steel, but heating of the processing solution to  $150 \pm 5^\circ\text{F}$  partially overcame this problem.

#### Stannous Solutions:

##### Pyrophosphate

A formula for a stannous pyrophosphate plating solution (8) was modified in order to tin coat, by immersion, trimetal assemblies. The composition of the modified solution was as follows:

- \* 50 grams  $\text{Sn}_2\text{P}_2\text{O}_7$
- 50 grams  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$
- 10 grams dextrine
- 1 gram gelatin
- Enough water to make a one liter solution.

The pH of the solution was 6.2. The solution was operated at  $195 \pm 5^\circ\text{F}$ . The trimetal assemblies were immersed in the solution for thirty minutes, water rinsed and air dried. There was a thick adherent shiny tin coating on the aluminum and steel of the trimetal assemblies (see Figure 1). There was a dark grey coating on the magnesium component.

The pH of the solution was still 6.1 after four or five processing cycles.

The resistance to corrosion, including galvanic corrosion, was better than that of the other coatings. Galvanic corrosion was first noticed after 2 hours exposure to salt spray.

This processing solution would coat any two metal combinations or aluminum and magnesium separately, but not steel separately.

- \* The  $\text{Sn}_2\text{P}_2\text{O}_7$  solution was prepared as follows:  
198 gms. of  $\text{SnCl}_2$  was added to 500 ml. of distilled water with constant agitation. Conc.  $\text{HCl}$  was added to the solution until all the  $\text{SnCl}_2$  was dissolved. This solution was poured into an aqueous solution of 196 gms. of  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$  and made up to one liter. The resultant solution was stirred and filtered by suction through a 41H filter paper. The precipitate was washed four or five times with distilled water and subsequently dried in an oven at  $230^\circ\text{F}$ .

Changes in the pH of the solution produces different coatings as follows:

pH 6.0 (by addition of  $H_3PO_4$  solution)

Aluminum - Conversion type coating  
Steel - Shiny tin coating  
Magnesium - Conversion type coating

pH 7.0 (by addition of 5% NaOH solution)

Aluminum - Very bright shiny tin coating  
Steel - Very bright shiny tin coating  
Magnesium - Thick adherent conversion type coating.

pH 8.0 (by addition of 5% NaOH)

Aluminum - Shiny tin coating  
Steel - Shiny tin coating  
Magnesium - Dull tin coating

#### Phosphate Solutions:

A zinc phosphating solution, that was accelerated and buffered to coat steel at room temperature, was used for coating the trimetal assemblies. It was found that if the phosphating solution was heated to  $200 \pm 5^\circ F$  it would phosphate coat all three of the steel grit blasted metals in trimetal assemblies. The processing time was thirty minutes. The phosphate coating on the magnesium metal had numerous grey spots. The addition of 3 - 5 grams  $AlCl_3$  per liter of solution either eliminated or decreased the size of the spots in subsequently produced phosphate coatings.

The phosphate coated trimetal assemblies were immersed in a near boiling five percent dichromate solution for ten minutes. There was no noticeable change in the coating.

It was necessary to adjust the pH of the phosphating solution after each processing of trimetal assemblies.

#### DISCUSSION

The 1100 aluminum sheet contained a minimum of 99 percent aluminum. The 5052 aluminum is an aluminum alloy containing magnesium as the major alloying metal. The magnesium sheet contained 3 percent aluminum and one percent zinc.

The stannate-chromate solution was a combination of the major chemicals found in some chemical processes for aluminum and magnesium. The solution coated the aluminum with a chromate conversion coating that appeared to be as good as a commercial chemical process for aluminum. The

solution coated the magnesium with an apparent tin coating that appeared to be as good as an immersion tin process for magnesium (see Figure 1b). The first solution developed would not satisfactorily coat the 1100 aluminum, probably because the galvanic potential between the 1100 aluminum and magnesium was not as great as the 5052 aluminum and magnesium. Modification of the solution eliminated this problem.

The phosphating of the trimetal assemblies produced a good paint base coating. The phosphate coatings did not improve the galvanic corrosion resistance of the trimetal assemblies. In fact, the phosphate coating in the presence of salt fog seemed to increase the galvanic corrosion. Examination of the phosphate coated trimetal assemblies, upon removal from salt fog, revealed a visual chemical reaction on the surface of the magnesium component.

The stannate-gluconate and the stannate-hypophosphite solutions produced good coatings on the trimetal assemblies (see Figure 1). The pH of the solutions had to be maintained within a narrow range, or the coatings would be inferior or no coatings were produced either on the magnesium or aluminum. Stannate solutions are not as stable as the following pyrophosphate solution.

The stannous pyrophosphate solution was very stable. The coatings adhered well to the metals (see Figure 1d). The solution coated trimetal assemblies over a wide pH range and coated well in recess areas. Coated trimetal assemblies exhibited good galvanic corrosion resistance. The pH of the solution can be maintained at neutral or near neutral (pH 7 to 8), thereby eliminating attacks on the metals in the trimetal assemblies during processing.

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illus., tables, (DA Project No. 1-H-O-24401-A-  
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blies were simultaneously coated in order to  
decrease galvanic corrosion and to develop  
coatings to be used for recoating structurally  
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