REPORT NO. NADC-83127-60

ALUMINUM-MANGANESE PLATING FROM A MOLTEN SALT BATH

E. J. Jankowsky
Aircraft and Crew Systems Technology Directorate
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974

15 AUGUST 1983

FINAL REPORT
AIRTASK NO. A320320A/001B/2F61542000
Work Unit No. ZM520

Approved For Public Release; Distribution Unlimited

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The report describes the scale-up of a molten salt bath for electroplating aluminum-manganese alloy coatings. Results of hydrogen embrittlement, adhesion, fatigue and corrosion tests of aluminum-manganese on steel are given.

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INTRODUCTION

There has been considerable effort to minimize the use of cadmium by Department of Defense activities because of its toxicity. While no single coating has been found to replace cadmium in all aircraft applications, aluminum has been found to be a good alternative coating material in many applications requiring good corrosion resistance and minimal effect on fatigue properties. Only two commercial aluminum coating processes, vacuum deposition and ion vapor deposition, have been developed to the point of being widely employed. Vacuum deposition has relatively poor covering power and adhesion is often only fair. Ion vapor deposition is proprietary and facilities for its application cannot fully meet the demand for the coating

This report describes an attempt to scale-up an aluminum-manganese plating bath that could produce an alternative to aluminum coating by vacuum processes. The work was performed under reference (a). The bath, which was originally developed by the National Steel Corporation, is described in references (b) and (c). It consists of a mixture of anhydrous aluminum chloride, manganese chloride, potassium chloride, and sodium chloride. This mixture is melted in a suitable vessel and operated at a temperature of 166-177°C (330-350°F). Plating is performed in the normal manner used for aqueous baths, the major difference being that the bath and the surrounding atmosphere must be kept as dry as possible. Anhydrous aluminum chloride reacts with water to form hydrochloric acid and aluminum hydroxy chlorides.

TEST METHODS

Bath Make-up and Operation

The bath was first made up with reagent grade chemicals in a one liter Teflon TFE beaker. Anodes were pure aluminum. The bath was fitted with a Teflon cover and heated by an electrical heating mantle. Operating temperature was measured with an ordinary mercury thermometer inserted through a hole in the lid. It was necessary to operate the bath under a laboratory exhaust hood because of the continual evaporation of aluminum chloride from the bath. Bath make-up was as follows:

Anhydrous aluminum chloride	79% by wt.
Anhydrous manganous chloride	1%
Sodium chloride	10%
Potassium chloride	10%

The bath was operated at current densities of 215-861 amperes per square meter (A/m^2) (20-80 amperes per square foot). Operating temperature was about 171°C (340°F). Analysis of deposits from this bath showed that the coatings contained about 25% manganese and 75% aluminum.

After successful operation of the small bath (sound, bright deposits), a 10-gallon bath with the same composition was made up in a glass-lined steel reactor vessel heated by a three zone electrical heating mantle. Temperature was controlled by a thermocouple connected to a standard relay system. The thermocouple was inserted in a closed Vycor tube and immersed in the bath. The top of the bath was covered with a tight-fitting Teflon lid with openings for the 1100 aluminum alloy anodes, stirring motor, thermocouples and plating. A loose-fitting Teflon lid was kept over the opening used for plating. The top of the bath was attached to a glove box that was kept dry by trays of dessicant inside the box as well as by a constant flow of dry nitrogen through the box. Additionally, air was occasionally fed into the glove box through a drying train by means of a small air pump. An exhaust tube was connected to the box into a fume hood. This bath was operated periodically for over two years on the original charge of chemicals. It was necessary only to make occasional additions based on analysis. The principal difficulty encountered was the large amount of white "smoke" (actually aluminum chloride) that filled the chamber when the loose cover was removed for plating. Much of the aluminum chloride vapor was prevented from entering the box by inserting the specimens as quickly as possible and then closing the opening on either side of the cathode connection with small Teflon covers.

In an attempt to improve the corrosion protection afforded by the coatings, a one liter bath was prepared, first with no manganese, and later, with enough manganese chloride added to make deposits containing 14% manganese.

Pretreatments

The following pretreatments were used for plating various specimens:

a. Glass bead blasting.

   1. Solvent degrease.

   2. Glass bead blast with fine glass beads.

b. Sulfuric acid etching.

   1. Alkaline clean anodically in a proprietary alkaline cleaner for steel ("Anodex") at six volts for one minute.

   2. Rinse in cold water.

   3. Etch anodically in 20% sulfuric acid solution for 1½ minutes at 538 A/m^2 (50 asf).

   4. Rinse.

   5. Dip in 25% hydrochloric acid, 15 seconds.

   6. Rinse in cold water.

   7. Dip in boiling distilled water.

   8. Dry with oil free hot air.

c. Electropolishing

   1. Alkaline clean anodically in Anodex six volts, one minute.

   2. Rinse.

3. Electropolish in the following solution for 10 minutes at 753 A/m^2 (70 asf):

Sulfuric Acid	15%
Phosphoric Acid	65%
Chromic Acid	10%
Temperature	52°C (125°F)

4. Rinse in cold water.

5. Dip in 25% hydrochloric acid, 15 seconds.

6. Rinse in cold water.

7. Dip in boiling distilled water.

8. Dry with hot air.

d. Grit blasting (titanium alloy only).

1. Solvent degrease.

2. Grit blast with aluminum oxide driven by dry argon gas in S. S. White blasting cabinet.

e. Deoxidizing (aluminum alloy only).

1. Clean in a proprietary non-etching cleaner (Oakite 164), five minutes, 160°F.

2. Rinse.

3. Deoxidize in a proprietary chromate type deoxidizer (Isoprep 188), five minutes, room temperature.

4. Rinse.

5. Dry.

Adhesion

Adhesion was determined by bending 1 mm (0.040") specimens 180° in a vise and examining the bend for flaking. More refined bend tests were conducted with 50.8 x 101.6 cm 1020 steel panels, 2.8 mm thick, in a universal testing machine. Specimens were all plated with 13 μm (0.0005") of aluminum-manganese and bent 180° around a 3.17 mm (1/8)" mandrel at a cross head speed of 1 cm (0.4") per minute. Adhesion was also determined by sectioning, mounting and polishing plated specimens and examining the bond metallographically at 100X.

Salt Spray Corrosion Tests of Screws

Steel screws from Navy stock were stripped of cadmium, recoated with 10 μm (0.0004") of aluminum-manganese and compared with screws coated with 10 μm of ion vapor deposited (IVD) aluminum furnished by the McDonnell Aircraft Company, and cadmium plated screws from stock (nominal thickness 7.6 μm (0.003") in SO_2 - salt spray.

The aluminum-manganese was applied from two one-liter baths, one that produced coatings with 25% manganese and one that resulted in deposits with 32% manganese. It was thought that higher manganese content would result in better corrosion resistance. All screws were chromated after plating. For corrosion testing, the screws were inserted in 7075-T6 aluminum alloy panels.

In addition to the screws tested in SO_2 - salt spray, six steel screws plated with 7.6 μm of aluminum-manganese and installed in 7075-T6 aluminum alloy plates were exposed for eleven months on the flight deck of the aircraft carrier, USS AMERICA. Similar assemblies were made up with cadmium-plated screws and ion vapor deposited aluminum coated screws and exposed on the flight deck.

Salt Spray Tests

Steel panels (1020 steel) were plated with 13 μm of aluminum manganese (25%) and 13 μm of aluminum manganese (14%), chromate conversion coated and exposed to 5% salt spray in 15° racks. A few panels were scribed through the coating to the basis metal prior to exposure.

Carrier Exposure Panels

Steel panels (4130 steel) were coated with 13 μm of aluminum manganese (25%), ion vapor deposited aluminum, and bright cyanide cadmium, respectively, and exposed on the flight deck of the aircraft carrier, USS AMERICA for eleven months. IVD aluminum coated panels were furnished by the McDonnell Aircraft Company. All the coatings were given a supplementary chromate conversion coating prior to exposure.

Hydrogen Embrittlement Tests

Round notched tensile specimens made of high strength AISI 4340 steel (1791-1929 MPa (260-280 ksi)) were used for sustained load testing. Notch dimensions were: root radius 0.10mm (0.004"), notch depth 1.016 mm (0.040") and diameter at the notch root 0.406 cm (0.160"). Specimens were pretreated by solvent degreasing and glass bead blasting. As plated specimens, were coated with 13 μm (0.0005") of aluminum-manganese and loaded in dead weight lever arm stress rupture machines within one hour after plating. The first specimen was loaded to 75% of the notched tensile strength and successively lower loads were applied to each newly plated specimen until a stress was reached at which no failure occurred in 500 hours. Plated and baked specimens were baked for 24 hours at 375°F following plating and before loading.

Fatigue Tests

Sheet flexure fatigue specimens of the type shown in Figure 1 were used for fatigue testing. Specimens were machined from AISI 4340 steel heat treated to a strength level of 1791-1929 MPa (260-280 ksi) All specimens, including the unplated controls, were cleaned anodically in an alkaline cleaner at six volts, rinsed in water, anodically etched in 20% sulfuric acid solution for 1½

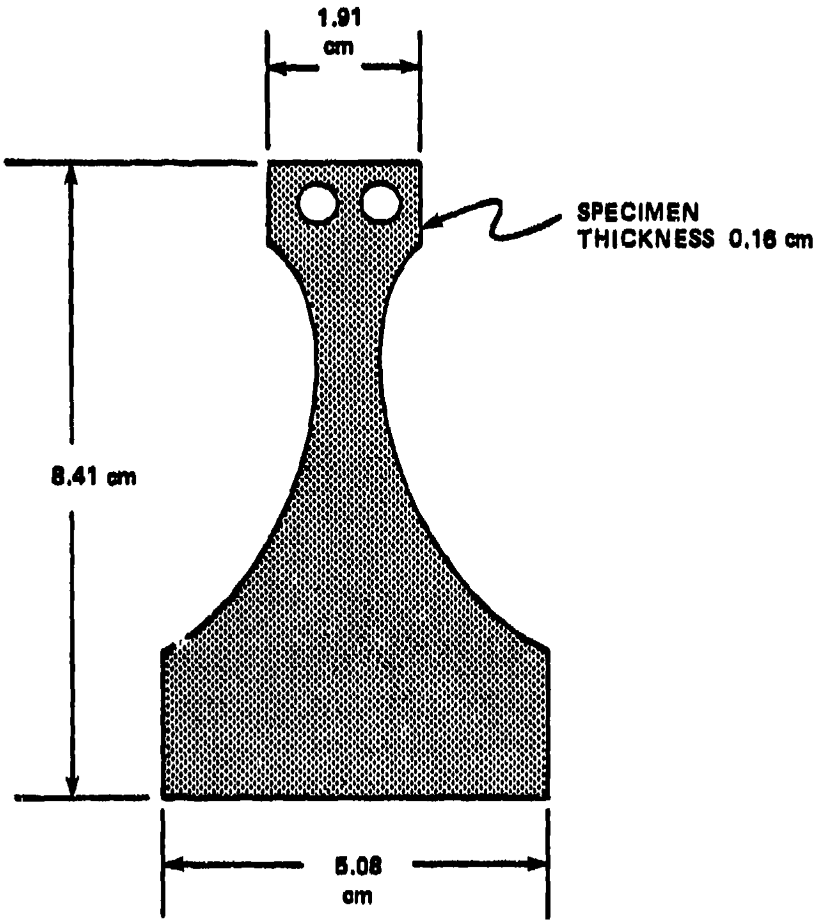

FIGURE 1. SHEET FLEXURE FATIGUE SPECIMEN

minutes at 538 A/m^2 (50 asf), rinsed, pumice scrubbed, rinsed, dipped in 25% hydrochloric acid, rinsed and dryed with a hot air blower. Specimens with 25% manganese were plated from the ten-gallon bath. Specimens with 14% manganese were plated from a one liter bath. All specimens were plated to a thickness of 13 μm (0.0005") including the bright cyanide cadmium plated specimens used for comparison.

Specimens that did not fail at the first stress applied were rerun at higher stresses to provide additional points on the high stress part of the fatigue curves.

Miscellaneous Testing

1. Plating Titanium Alloy

Specimens made of 6 A1-4V titanium alloy were pretreated by grit blasting in argon before plating. Adhesion was determined by bend test and metallographic examination.

2. Plating Aluminum Alloys

Specimens of 6061 and 7075 aluminum alloy were pretreated by non-etch cleaning and deoxidizing, plated with 13 μm (0.0005") of aluminum manganese (25%) and bent in a vise to measure adhesion qualitatively. A few specimens were also examined metallographically to determine the quality of the bond.

3. Anodizing

An attempt was made to anodize the aluminum-manganese coating in a standard 15% by weight sulfuric acid bath.

4. Chemical Film Treatment

Chemical conversion coatings were applied to the aluminum manganese coatings in accordance with MIL-C-5541 immediately after plating and rinsing.

RESULTS AND DISCUSSION

BATH MAKE-UP AND OPERATION

The principal difficulty in the operation of the molten salt aluminum plating bath is the evaporation of aluminum chloride. The amount of aluminum chloride given off does not change the composition of the large bath very rapidly, but it does create problems with both visibility and deterioration of materials. When the opening in the lid is uncovered for insertion of a specimen, clouds of aluminum chloride fill the glove box. If it were not for the air pump pushing out the "smoke," it would be extremely difficult to see well enough to operate the bath after the first specimen was introduced. Additionally, the aluminum chloride forms hydrochloric acid when any water vapor is present and is extremely corrosive to many materials. For example, an ordinary laboratory stirring motor was used inside the glove box for agitating the bath. After about six months of use, the motor failed completely.

As corrosive as it is, the aluminum chloride did not appear to be causing any health problems and was easily contained in the glove box. However, self-contained breathing equipment was used when the aluminum chloride was transferred from the shipping container to the reactor vessel during make-up of the 10-gallon bath.

The bath operates at fairly low temperatures (157-177°C), has good conductivity and plates at reasonable current densities (215-861 A/m^2 (20-80 asf). A current density of 430 A/m^2 (40 asf) was used for most of the specimens. Deposits formed at that current density were usually matte white to bright. Brighter deposits were obtained at higher current densities, but there was a greater tendency for rough, burned deposits to form on panel edges.

PRETREATMENTS AND ADHESION

Results of tests of panels bent in a vise indicated that any of the methods employed produced good adhesion on steel. The simplest method, glass bead blasting, has the advantage of not requiring drying following cleaning, but subsequent testing in a universal testing machine showed only fair adhesion for this method. Both sulfuric acid etching and electropolishing gave excellent results, except for one electropolished specimen. This specimen failed in the bend tests for unknown reasons. Possibly, the oxide reformed on the specimen between electropolishing and drying. Metallographic examination of the three methods for pretreating steel indicated that both sulfuric acid etching and electropolishing resulted in a good bond while glass bead blasting gave only fair adhesion.

The titanium specimens were surprisingly easy to plate following grit blasting. No special precautions were taken except for using dry argon to propel the grit rather than compressed air. The bond was very good as confirmed by metallographic examination, and adhesion was consistently excellent.

Results with aluminum alloys were not as consistent. Aluminum alloy 6061 could be plated with good adhesion by degreasing, deoxidizing, and drying. However, this same pretreatment system resulted in very inconsistent adhesion on 7075-T6 aluminum alloy.

SALT SPRAY CORROSION TESTS OF SCREWS

Early test results with plated screws in 7075-T6 aluminum panels showed the coating with 25% manganese to be more resistant to corrosion than the 32% manganese coating and slightly better than IVD aluminum coating in SO_2 salt spray. Results of these are given in Table I.

CARRIER EXPOSURE RESULTS

Aluminum-manganese (25%) panels exhibited corrosion behavior similar to that obtained in salt spray. Within two months, rust spots appeared on the panels, but rusting progressed slowly. At the end of the exposure period, eleven months, the coating was 90% replaced by rust. Coatings of the same thickness (13 μm) of ion vapor deposited aluminum showed general white corrosion after two months of exposure, but no rust spots. There was still only white corrosion after four months. After seven months, the panels were 75% rusted and by the end of nine months, 100% rusted.

Carrier exposure tests of aluminum manganese, ion vapor deposited and cadmium plated screws in 7075-T6 aluminum alloy plates indicated that cadmium resulted in the least corrosion in the countersink area of the aluminum alloy. Aluminum-manganese protected the aluminum countersink areas slightly better than ion vapor deposited aluminum and showed less rusting on the screw heads. This was an unexpected result. It was thought that pure aluminum (IVD) would offer the best protection to the countersinks.

TABLE I. PLATED SCREWS IN 7075-T6 ALUMINUM ALLOY PANELS

RESULTS OF SO_2 SALT SPRAY TESTS

	First Rust		Appearance at 288 Hours -
*Coating	Drive Slots	Head	End of Test
Al-Mn (25%)	72 hrs	216 hrs	Corrosion progressing from drive slots Coatings still intact
Al-Mn (32%)	72 hrs	72 hrs	Corrosion on all heads Worse in drive slots
IVD A1	-	72 hrs	Much rust on heads
Cadmium	-	48 hrs	All rust on heads. Very bad

*Four screws per coating.
All coatings given a supplementary chromate treatment.

FATIGUE TEST RESULTS

Fatigue test results are given in Figure 2 and Table II. Aluminum-manganese coatings with 25% manganese resulted in a 19% loss in fatigue strength of 4340 steel sheet flexure specimens compared with a 10% loss for cadmium plated specimens and a 7% loss for aluminum-manganese with 14% manganese.

SALT SPRAY CORROSION TEST RESULTS

Steel panels with chromated aluminum-manganese (25%) coatings, 13 μm (0.0005") thick showed rust spots after 72 hours of exposure to 5% salt spray. However, rusting progressed very slowly and there was little change in the appearance of the panels for two months (1440 hours), at which time testing was discontinued. Panels coated with chromated 14% manganese alloy (13 μm) were not corroded after 2000 hours of exposure. Coatings exposed without a supplementary chromate conversion coating did not protect as well as those with a chromate coating. Rust stains appeared on the unchromated 25% manganese coatings in 24 hours.

Scribed panels of both 25% manganese and 14% manganese performed as expected. The steel rusted in the scribes in 24 hours with the higher manganese coating. Scribed 14% manganese coatings were not rusted in the scribes in 144 hours when testing was discontinued because rust spots began to form on the top edge of the panel. This panel had a coating only 7.6 μm thick as opposed to the unscribed panels which were coated with 13 μm. Panels coated with 7.6 μm (0.0003") of pure aluminum (no manganese) showed much white corrosion after 24 hours and almost complete rusting of the faces in 144 hours.

From the corrosion tests, it is apparent that 25% manganese coatings protect by barrier action while 14% manganese coatings are sacrificial to steel.

HYDROGEN EMBRITTLEMENT

Hydrogen embrittlement test results, given in Table III, indicate that the molten salt plating bath is very embrittling to high strength steels. Recovery was apparently very complete when plated specimens were baked for 24 hours at 191°C (375°F).

ANODIZING RESULTS

Attempts to anodize 25% manganese coatings were unsuccessful even when the substrate was an aluminum alloy. Instead of the normal reduction in current as the anodic film formed, the current remained high and eventually the aluminum wire holding the specimen burned through. Regardless of the manner in which the current was applied, no anodic film appeared to form.

CHEMICAL FILM TREATMENT

Chromate conversion coatings (MIL-C-5541) could be applied to both 25% and 14% manganese coatings without difficulty. The coating was much darker on the higher manganese alloy and appeared to form more slowly. The 14% manganese alloy accepted the treatment very well and the film had the same golden brown color it has on most aluminum alloys.

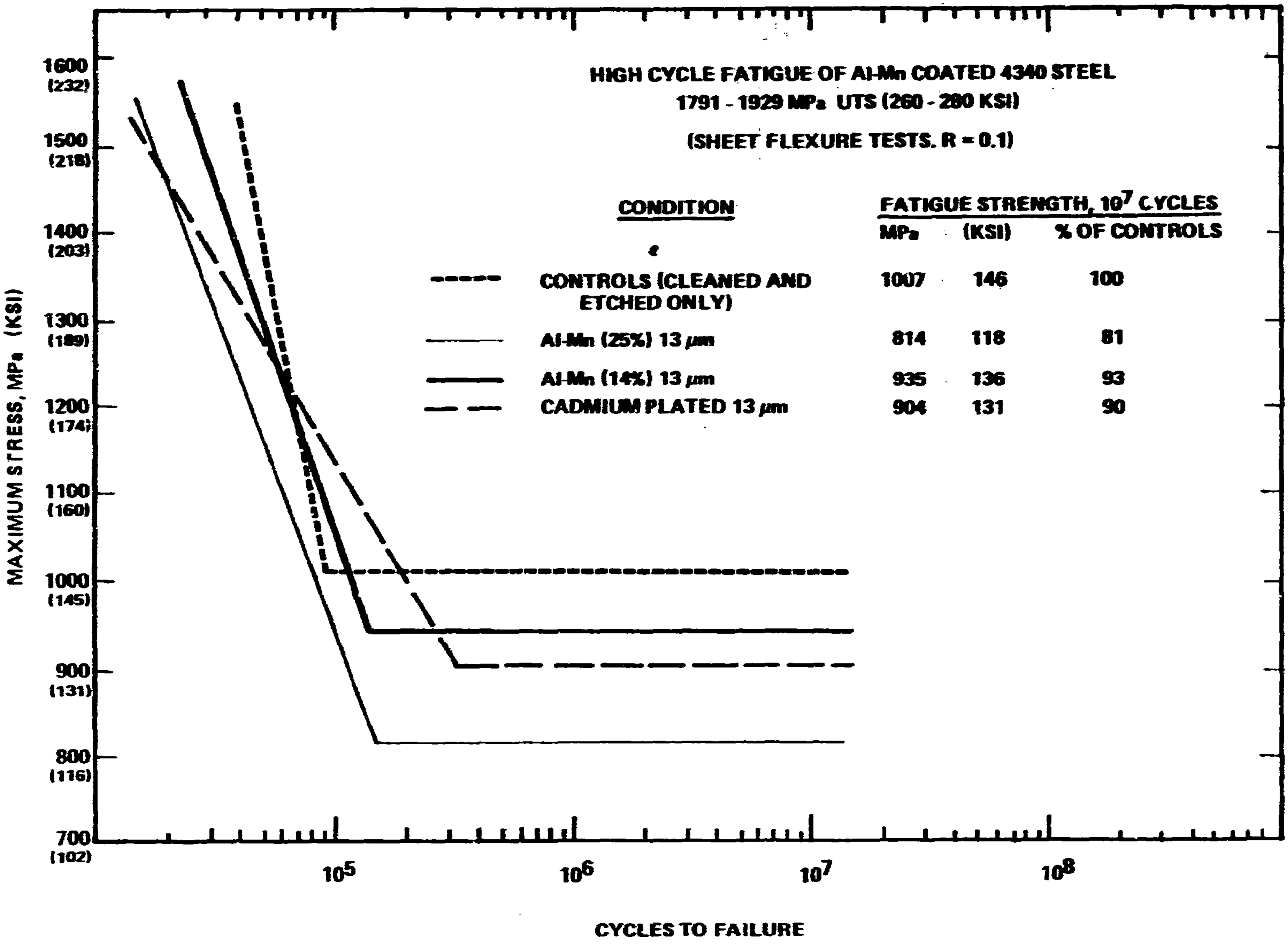

FIGURE 2. HIGH CYCLE FATIGUE OF Al-Mn 4340 STEEL

TABLE II. SHEET FLEXURE FATIGUE RESULTS

Control Specimens, 4340 Steel,
1791-1929 MPa (260-280 ksi), Ratio = 0.1

Specimen No.	Stress, MPa (ksi)	Total Cycles
3	723(105)	14,819,400 NF *
4	792(115)	12,491,400 NF
5	826(120)	19,926,400 NF
6	847(123)	15,169,200 NF
2	861(125)	121,600 **
7	875(127)	18,113,300 NF
8	930(135)	18,542,800 NF
9	978(142)	27,844,900 NF
1	1034(150)	51,200
Reruns		
6	1034(150)	421,300
5	1102(160)	83,300
3	1137(165)	37,200
4	1137(165)	66,900
8	1240(180)	65,700
9	1378(200)	59,100
7	1447(210)	62,800

Aluminum-Manganese (25%) Plated Specimens
Coating Thickness = 13 μm (.0005")

Specimen No.	Stress, MPa (ksi)	Total Cycles
12	723(105)	10,207,700 NF *
13	758(110)	19,855,100 NF
15	779(113)	15,235,200 NF
11	792(115)	149,400 ***
16	806(117)	20,559,900 NF
18	806(117)	10,254,400 NF
17	827(120)	140,900
10	861(125)	172,700
14	1034(150)	65,100
Reruns		
12	1034(150)	70,300
13	1102(160)	79,200
15	1137(165)	20,000
18	1309(190)	37,100
16	1516(220)	33,200

* NF = No failure - Run out
** Failed at surface flaw
*** Failed at one edge

TABLE II. SHEET FLEXURE FATIGUE RESULTS (CONT'D)

Aluminum Manganese (14%) Plated Specimens
Coating Thickness = 13 μm (.0005")

Specimen No.	Stress, MPa (ksi)	Total Cycles
31	875(127)	17,830,700 NF *
28	896(130)	17,265,600 NF
32	916(133)	13,009,600 NF
33	930(135)	10,156,500 NF
30	944(137	191,700
32	965(140)	38,100
29	999(145)	77,100
27	1102(160)	83,500
34	1275(185)	81,100
Reruns		
28	1378(200)	36,000
31	1447(210)	25,400
32	1516(220)	22
33	1585(230)	28,000

Bright Cyanide Cadmium Plated Specimens
Coating Thickness = 13 μm (.0005")

Specimen No.	Stress, MPa (ksi)	Total Cycles
24	861(125)	10,238,100 NF *
26	882(128)	25,805,000 NF
25	930(135)	4,000,000
23	930(135)	333,200
19	965(140)	10,133,600 NF
22	1034(150)	158,300
21	1102(160)	123,300
20	1240(180)	49,100
Reruns		
19	1240(180)	100,300
24	1378(200)	45,700
26	1557(226)	24,100

* NF = No failure - Run out

TABLE III. HYDROGEN EMBRITTLEMENT TEST RESULTS WITH ALUMINUM-MANGANESE PLATED NOTCHED TENSILE SPECIMENS (AISI-4340 STEEL, 1791-1929 MPa STRENGTH LEVEL).

I. As Plated – 13 μm (0.0005")

Stress (% NTS)	Time to Failure (hrs)
75	5.6
75	2.9
75	15.2
65	2.8
55	2.9
45	3.6
35	316.2
25	571.1 *

II. Plated and Baked 24 hours at 191°C (375°F)

Stress (% NTS)	Time to Failure (hrs)
65	505.6 *
75	505.7 *
75	500.0+ *
75	500.0+ *
85	500.0+ *

* Did not fail. Test discontinued.

CONCLUSIONS

1. Because of the large amounts of aluminum chloride vapors evolved by the bath, the molten salt aluminum-manganese bath is difficult to operate.

2. The bath is not difficult to maintain or to keep dry enough to perform satisfactorily.

3. Coatings with 14% manganese offer better protection to steel than 25% manganese coatings.

4. The loss in fatigue properties from 14% manganese coatings is very small.

5. Adherent coatings can be obtained from the aluminum-manganese bath by proper surface preparation.

6. The aluminum-manganese bath plates titanium alloy very well with very simple surface preparation.

RECOMMENDATIONS

It is recommended that no further work be conducted on the aluminum-manganese molten salt bath because of the difficulty in operating baths of this type. While it is recognized that 14% manganese deposits possess many of the desirable properties that were sought at the beginning of this work, the Environmental Protection Agency has relaxed its original stand on the use of cadmium and is no longer attempting to eliminate cadmium completely. The need for a substitute is therefore less pressing than it was when the work was initiated.

ACKNOWLEDGEMENT

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