ELECTROLYTIC COATINGS FOR

MAGNESIUM ALLOYS

By

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PREFACE

The selection of a protective coating for magnesium is dependent on many factors such as its stability under saline conditions, bonding strength, dielectric strength, abrasive resistance, etc. This work is concerned only with the stability of coatings under saline conditions. Interested parties should consult the literature, for other property evaluations, prior to the selection of any one coating process.

The author wishes to express his appreciation to Dr. Charles L. Nickolls for his counsel on this project and to other members of the Chemical Engineering Department for their encouragement and interest. The author desires to acknowledge use of the facilities of the Oklahoma Institute of Technology, and in particular the Electrical Engineering Department for use of special electrical gear.

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Lastly, the author is indebted to his loving wife Martha, for without her thoughtfulness, this work could not have been completed.

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INTRODUCTION

The development of satisfactory protective coatings for use on magnesium alloys has come about as a result of the modern need for light structural materials. Magnesium alone displays good corrosion resistance, however pure magnesium has poor structural properties. Aluminum is one of the principle alloying agents for magnesium. The addition of this element greatly increases the new made alloy's susceptability to corrosive attack. Manganese is generally added to the alloy in proportion to the aluminum present since it has a property which tends to reduce the deleterious effect of the aluminum. Thus far, no one has determined a ratio of manganese to aluminum that will completely compensate for the aluminum's tendency to accelerate corrosion. Other elements such as iron also greatly accelerate the corrosion if present in excess of established critical maximum values. Consequently, the usefulness of magnesium alloys is in some cases limited by the quality of their protective coatings.

Through the years there has been in excess of one hundred different methods proposed for surface protecting magnesium alloys. The greater number of these methods has been purely chemical in nature. Since the first electrochemical method was suggested in 1926, this method has come to be regarded as superior to other methods. The reason for this is due to the outstanding qualities of many of the new protective coatings made possible by the electrochemical methods.

The objective of this work is to make an evaluation of four of the newer electrochemical methods for producing coatings on magnesium alloys. In particular this work is aimed at determining over what latitude the inventor's recommended current density and time requirements may be varied and still maintain a serviceable protective film. The protective coatings studied are the Dow #12 Caustic Anodize for magnesium, the Dow #14 a.c. Anodic Coating for magnesium, the Dow #17 Anodize for magnesium, and the HAE Coating for magnesium. In addition a chilled 15% by weight solution of sulfuric acid is investigated for possible film forming properties. A method of coating magnesium proposed by the National Bureau of Standards Journal of Research is briefly reviewed.

The results of this work may be of particular interest to the industrial firms making use of these treating processes. It is the belief of the writer that from the results of this work it may now be possible to determine optimum operating conditions for those cases where it is now of questionable economic feasibility to follow exactly the recommended treating cycles. The work is of academic interest for the effects of current density on coating quality.

 $\mathbf{2}$

TABLE 1

THE DEVELOPMENT OF ANODIC COATINGS FOR MAGNESIUM ALLOYS

Process Name or Principle Electrolyte Reagents	Date Appearing in the Literature	Likely Film Components
Hydrogen Fluoride	1926	Fluoride, oxy-fluoride
National Bureau of Standard	l s 1936	Oxide, phosphate, and chromate
Sodium Hydroxide (5%)	1946	Oxide, hydroxide
Manodyz Process	1947	Oxide, silicate
Sodium Carbonate	1948	Carbonate
Dow Chemical #12 Process	Prior to 1952	Oxide, hydroxide
Dow Chemical #14 Process	Prior to 1952	Oxide, silicate, and borate
HAE Process	1951	Oxide, mánganate, and hydroxide
Ammonium sulfate, sodium dichromate and ammonium hydroxide	1952	Oxide, sulfate, and chromate.
Lithium Hydroxide and Diethylene Glycol	1952	Oxide, hydroxide
Dow Chemical #17 Anodize	1953	Fluoride, chromate

) .

PROCEDURE

Cleaning of Test Panels:

Test panels of magnesium alloy FS-1 were furnished by the Dow Chemical Company. These panels were cut into sections 2 3/4 by 4 inches from 20 gage sheets of the alloy. The sheets had been given a mill dichromate treatment and a coat of heavy oil had been applied to reduce any tendency to corrode while the sheets were in storage. It was necessary to clean the panels down to the bare metal before the coatings to be evaluated could be applied. The following cleaning scheme was used. This scheme is a combination of several suggestions from the literature.¹²

The panels were submitted to an alkaline electrolytic cleaning step. This was accomplished by making the panels the cathode in a bath composed of 3 ounces of sodium carbonate, 2 ounces of sodium hydroxide and 1/2 ounce of detergent per gallon of solution. A direct current under a potential of 12 volts was impressed on the panels. The current density was maintained at approximately 20 amperes per square foot by inserting a slide resistor in series with the panels. The temperature of the bath was maintained in the range 190-212 °F. by the use of an electric hot plate under the stainless steel cleaning tank. The panels were kept in this bath for nearly 10 minutes or until no water break was noticed when the panels were withdrawn from the bath.

Following the alkaline cleaning the panels were rinsed in tap water and then immersed for 1 minute in a 24 ounce per gallon

solution of hot chromic acid. This bath removed the last traces of the oil and dichromate films on the test panels. The panels were again rinsed in tap water and then immersed in a 2% solution of nitric acid. The nitric acid pickle was effective in giving the panels a brighter finish and removed any surface impurities that may have been rolled into the sheets. Following the nitric acid pickle the panels were washed by hand in a warm solution of "Fab" detergent, throughly rinsed and allowed to air dry.

Identification of the individual test panels throughout the work was accomplished by a notch and hole system. See page 21 in the Appendix for complete details.

Application of the Dow #12 (Caustic Anodize) Coating:

The procedure for the application of the Dow #12 coating was that recommended in the Dow <u>Magnesium Finishing</u> handbook.¹² The current density and time of application were varied between 25 and 200% of the recommended maximum values. This gave a range of actual current densities from 5-40 amperes per square foot and a range of treatment times from 6.25-50 minutes. The application of this coating was comparatively easy to control since this is not a selfsealing coating. That is to say, the current density once set, remained constant throughout the run without subsequent adjustment of the voltage.

Following the anodizing in a bath of the composition shown in Table 3 , the panels were thoroughly rinsed in tap water and then immersed for 5 minutes in a neutralizing bath. The composition of this bath is indicated in Table 4 . The panels were rinsed once

again after neutralization and allowed to air dry. See Figure 7 for the flow chart of the process and Figure 6 for the equipment arrangement.

Application of the Dow #14 a.c. Anodic Coating:

The procedure for the application of the Dow #14 coating was that recommended in the Dow Magnesium Finishing handbook. The current density and time of application were varied between 25 and 200% of the recommended maximum values. This gave a range of actual current densities from 3.75 to 30 amperes per square foot and a range of treatment times from 5 to 40 minutes. This coating is of the self-sealing type. That is, as the coating builds up on the surface of the panels, the current density falls off. To hold the current density constant for a run it was necessary to continually adjust the voltage across the work. This was done by changing the position of the armature in the transtat and by changing the setting of the series resistor. The voltage across the work rises from 0 to about 50 volts in a fraction of a second. The voltage continues to rise rapidly to about 75 volts and then gradually levels off. Maximum voltage for any run in this work was 128 volts. See Figure g for the flow chart of the process, Figure 6 for the equipment arrangement and Table 5 for the composition of the bath. Application of the Dow #17 Anodize for Magnesium:

The procedure for the application of the Dow #17 Anodize for magnesium was that recommended in the literature.¹¹ The current density and time of application were varied in a different manner than was the case for the other coatings. In this case the recommended conditions were expressed in ampere-minutes of treatment

time. The specified range was from 200-500 ampere-minutes per square foot of surface. The actual range over which the tests were run in this work was from 125-1000 ampere-minutes per square foot. This range represents from 25 to 200% of the maximum recommended values. This coating is also of the self-sealing type and consequently this required the adjustment of voltage throughout the runs. The voltage in this case rises quickly to 40 volts and then rapidly to about 70 volts. From this point the voltage rise is rather slow with a maximum of about 92 being reached. See Figure 9 for the flow chart of the process, Figure 6 for the equipment arrangement and Table 6 for the composition of the bath. Application of the HAE Coating for Magnesium:

The procedure for the application of the HAE Coating for magnesium was that recommended in the literature.²⁶ The current density and time of application were varied from 25 to 200% of the recommended maximum values. This gave a range of current densities from 3.75 to 30 amperes per square foot and a range of treatment times from 22.5 to 180 minutes. This coating is also of the selfsealing type and required constant adjustment of the voltage in order to maintain the desired current density. The voltage in this case rises quickly to 40 volts and then rapidly to about 70 volts. From this point the voltage rise is rather slow with a maximum of about 93 volts being attained. See Figure 10 for the flow chart of the process, Figure 6 for the equipment arrangement and Table 2 for the composition of the bath.

Evaluation of the Coatings:

The evaluation of a protective coating is an extremely difficult task at the best. Many schemes have been suggested for the evaluation of the coatings, each with its own particular applications and limitations. The method selected for this work is one that is an old standard in the coatings field, that being the exposure of the coating to a 20% salt fog.

A salt fog chamber with racking to accomodate 105 test panels was assembled and used to evaluate the coatings for this project. The chamber was a lead lined, open topped tank. Eight wooden racks were fashioned with slots at 15° from the vertical to hold the panels. A salt fog generator was made by assembling the points of a #16 and a #18 hypodermic needle at right angles to each other. By passing air at about 12 psig through the #18 needle a syphon was generated in the #16 needle. A flow of 20% salt solution was thus syphoned into the jetting air stream from a reservoir connected to the #16 needle by a rubber hose. This flow of salt solution in combination with the jet of air produced a very fine mist or salt fog. A bleeding of low pressure steam into the chamber was used in holding the temperature of the chamber between 95-105°F. The tank was covered over with a sheet of polyethlene, this prevented the fog from escaping into the room and allowed limited visual inspection of the chamber. A water jet was used to remove condensate and residue from the bottom of the tank.

One set of coated panels was submitted to 200 hours of salt fog exposure. Following exposure, the panels were rated for overall appearance by three fellow students. The results of these ratings are

included in Tables 15-18. The panels were then scrubbed with a soft bristle brush to remove loose corrosion products. Photographs of the scrubbed corroded panels are presented in Figure 14.

Estimation of the percent of coating failure was made in the following manner. A count was made of the corrosion pits on those panels having less than about 3% failure. By calculating the average size of a corrosion pit, multiplying by the number of pits and then dividing by the surface area, the percent failure was attained. Three independent estimations of the percent failure were weighed in estimating the failure for those cases where the figure was in excess of about 3%.

The results of the percent of coating failure are plotted against the ampere-minutes of treatment per square foot. Different symbols for the various current densities were used in preparing these charts which are listed as Figures 1-4.

There has been no direct correlation of the results of salt fog exposure with other types of weathering of coatings. In order that there might be some comparison drawn for one other type of exposure at least, duplicate panels of all runs in this work are being evaluated at the Dow Chemical Company plant site in Freeport, Texas. The results of the outdoor weathering are not necessary in completing the work at hand, but the results will be of significant value in aiding the correlation of salt fog and outdoor exposure results.

Evaluation of Sulfuric Acid for Coating Properties:

Recently, Patent No. 2692851 was awarded to Burrows of Alcoa for a new method of coating aluminum. This method involves the anodization of the metal in a chilled 15% by weight solution of sulfuric acid for 90 minutes with a current density of 20-25 amperes per square foot of surface area. An attempt is made to treat an alloy of magnesium in the same manner.

One and one-fourth pounds of 96% sulfuric acid are diluted with 7.08 pounds of water to prepare a 15% by weight solution of the acid. This solution, contained in a four liter beaker was centered in a five gallon bucket and packed in place with crushed ice and rock salt. After two and one half hours the temperature of the solution was reduced to 30°F.

A 12 volt d.c. power source was utilized in providing a current flow of 4.16 amperes. This current flow is equivalent to 25 amperes per square foot of panel surface. A 2 3/4 by 4 inch panel was made the anode and a carbon electrode was made the cathode in the solution. Review of A Magnesium Anodize Suggested by the Bureau of Standards:

A water solution composed of 10% by weight of sodium phosphate (NaH₂PO₄ : H₂O), 4% by weight of sodium dichromate (Na₂Cr₂O₇ : 2H₂O), and phosphoric acid to control the Ph at 4.0 - 4.8 was prepared. Several test panels were prepared using the recommended current densities.⁸ The coating is not of the self-sealing type, and is reported to be very susceptible to corrosive attack under saline conditions.⁸ The color of the film when properly formed is dirty green to shiny black. The film has very poor adherence to the base metal and flakes off the compression side of the panel even on a 3/4 in. radius bend.

RESULTS

An evaluation of four industrial anodic coatings for magnesium has been made by exposing test panels to a 20% salt fog atmosphere. The particular conditions of current density and time of treatment have been designed to determine over what latitude the recommended treatments might be varied without sacrificing coating quality; and to determine what effect, if any, current density has on the coating. The conclusions and reasoning for these conclusions follow.

The Dow #12 Caustic Anodize:

This coating was found to be not of the self-sealing type. The current density remained constant once the initial voltage had been set. A study of Figure 1 reveals that the two families of points for less than the recommended current densities lie to the right of the curve. This indicates that less than the recommended current densities are not effective in building up the protective coating and should be avoided. Fifty percent less than the specified treatment of 500 ampereminutes per square foot gives a good degree of protection. Panels given a treatment of 250 ampere-minutes per square foot should develop only about .6% coating failure after 200 hours in a 20% salt fog atmosphere. This compares with .38% for the recommended treatment. Over treatment reduces the percent of failure at 200 hours, but in view of the added application cost and small percent of failure at the specified level, it is likely that over treatment would be unwarranted. This would be especially true if a sealant were to be applied to the coated panel.





The Dow #14 Anodize:

This coating was found to be of the self-sealing type. As a result it was necessary to make periodic adjustments of the voltage in order to maintain the constant level of current density. A study of Figure 2 indicates that any treatment less than that recommended, regardless of current density, gives an inferior coating. This is evidenced by the fact that points representing less than the recommended treatment for all current densities lie far to the right of the zone of recommended treatment. An even distribution of various current density points seems to indicate that a variation from the recommended current density has no effect on the coating quality. Treatments in excess of that specified gave slightly superior protection. The results of this work indicate a treatment of 300 ampere-minutes per square foot should provide a coating having only about .7% failure after 200 hours in a 20% salt fog atmosphere. A treatment of 1000 ampere-minutes should provide a coating having only about .3% failure.

The Dow #17 Anodize:

The Dow #17 Anodize was found to be of the self-sealing type. It too required periodic adjustments of the voltage in order to maintain the desired current density. This coating seems to be the best of the Dow coatings. Current density had no detectible effect on the coating quality and less than the recommended treatment gave excellent protection. A study of Figure 3 indicates panels receiving 125 ampere-minutes treatment per square foot had only about .25% failure compared to .1% failure for the panels receiving the recommended treatment of 500 ampere minutes per square foot. Over treatment reduces the percent of failure to some degree. A panel having a treatment of 1000 ampere-minutes

The Effect of Salt Fog on the Dow #14 Coated Panels



Current Density 30 Amp/sq ft Current Density 22.5 Amp/sq ft
 x Current Density 15 Amp/sq ft x Current Density 7.5 Amp/sq ft A

Current Density 5 Amp/sq ft .

(Recommended treatment 300 ampere-minutes at 15 ampere/square foot)







Current Density 100 Amps/sq ft
 Current Density 75 Amps/sq ft
 Current Density 50 Amps/sq ft
 Current Density 25 Amps/sq ft
 Current Density 12.5 Amps/sq ft

(Recommended treatment 500 ampere-minutes per square foot, no specified current density) should have only about .08% failure in 200 hours. This small percentage compares to about 10 corrosion pits, 1/32 inch in diameter on the surface of a 2 3/4 by 4 inch panel. The power consumed in application of the recommended treatment was determined by graphical integration of the voltage rise curve in Figure 20. This value was calculated to be .665 Kw-Hr/Sq Ft.

The HAE Coating:

The HAE Coating for magnesium was also found to be of the selfsealing type. This coating provides superior protection over a wide range of treatments as did the Dow #17 Anodize. The panels receiving the recommended treatment of 1350 ampere-minutes per square foot developed only about .16% failure after 200 hours in the fog chamber. Less than the recommended treatment gave a good degree of protection over a wide range. Panels receiving only 350 ampere-minutes treatment per square foot had but .8% failure. Over treatment gave increased protection with a minimum of failure being .021%. This percentage compares to only 3 corrosion pits about 1/32 inch in diameter on the surface of the panel. Considerable variation from the recommended current density may give a lower degree of protection. This is in evidence by the fact that the family of points for the recommended current density are slightly to the left of the curve in Figure 4. A better explanation of this observation would probably lie with the condition of the panel surfaces prior to treating. Even though all panels were subjected to the same cleaning scheme, it is possible that this particular group of panels may have had fewer surface inclusions to affect the finished coating. In consideration of the very small percent of failure observed, this appears to be the preferred explanation.

The Effect of Salt Fog on the HAE Coated Panels



Current Density 15 Amp/sq ft

- × Current Density 7.5 Amp/sq ft Current Density 5 Amp/sq ft Δ

1350 ampere-minutes at 15 ampere/square foot)

The power consumed in application of the HAE Coating, calculated from Figure 19, was found to be 1.505 Kw-Hr/Sq ft.

Sulfuric Acid as an Electolyte for Anodizing Magnesium:

Chilled sulfuric acid of the concentration tested is of no value in providing a protective coating for a magnesium alloy. As soon as the magnesium panels were immersed in the solution, a violent evolution of hydrogen began. The flow of current through the bath had no apparent effect in either increasing or decreasing the evolution of the hydrogen. After one minute in the bath a panel of .0325 inches in thickness was decreased to .0037 inches in thickness. Subsequent attempts to form any sort of a coating in this bath were without success. The results of this work are quite in line with what might be expected on analysis of the likely film products. The sulfates of both aluminum and magnesium are soluble so the likelihood of them being present to any extent in a film is rather small. Likewise the hydroxides of both these elements are soluble in acid. This leaves only the oxides of the metals for film formation. The oxide film of aluminum is self-sealing since its oxide occupies 1.24 times the space of the metal from which it is derived.²² The case of magnesium is different, here no seal can be attained since the oxide of magnesium occupies only .79 times as much space as did the metal from which it was derived. The evolution of the hydrogen from the magnesium is so violent that any oxide formed would be carried into the solution. Higher concentrations of acid might be used in conjunction with lower temperatures. Lower temperatures would necessitate extensive refrigeration and probably prohibit such a process because of the added cost.

BIBLIOGRAPHY

- 1. American Society for Testing Materials, Bulletin B-117-44t, 1948.
- 2. Barbian, H. A., "Finishing Systems For Magnesium", <u>Materials</u> and Methods, v 39, p 102-5, January 1954.
- 3. Barbian, H. A., "How to Finish Magnesium", Modern Metals, December 1953.
- 4. Black, G., "Anodizing of Magnesium Alloys for Protection and Appearance," Product Engineering, v 18, p 122-4, May 1947.
- 5. Bleineis and Fusco, "Protective Treatments for Magnesium," Metals and Alloys, v 21, p 417, February 1945.
- 6. Boyer, J. A., "Corrosion of Magnesium and Magnesium Alloys Containing Manganese," Report No. 248, <u>National Advisory</u> Committee for Aeronautics, 1926.
- 7. Brooks and Perkins, Inc., "HAE Finish for Magnesium," trade bulletin, undated, Detroit 16, Michigan.
- Buzzard and Wilson, "Anodic Coatings of Magnesium Alloys," U. S. Bureau of Standards Journal of Research, v XVIII, p 83, October 19, 1936.
- 9. DeLong, H. K., "New Anodic Coating for Magnesium," Modern Metals, v 9, n 2, p 90-92, March 2, 1953.
- DeLong, H. K., "Electroplating on Magnesium," Paper presented at the Institute of Metal Finishing, spring conference, Hotel Majestic, Harrogate, April 23, 1953.
- 11. Dow Chemical Company, "Dow No. 17 Anodize for Magnesium," Bulletin No. DM 141 3M-254, undated.
- 12. Dow Chemical Company, "Magnesium Finishing", Bulletin No. DM 95A, undated.
- 13. Evangelides, Harry A., "A New Finish for Magnesium Alloys," Reprint from Organic Finishing, October 1951
- 14. Evangelides, Harry A., "HAE Process Application Characteristics" Light Metal Age, v 12, p 12-13, April 1954.
- 15. Evangelides, H. A. and Frager, M., "Getting the Most From HAE Coatings," Metal Progress, v 62, p 81-4, September 1952; also Production Engineer, v 23, p 278-280, December 1952.

- 16. Gillespie, R. G., "Properties of HAE Finish for Magnesium," Materials and Methods, v 38, p 104-5, November 1953.
- Gray, A. G., "Recent Developments in Protective Coatings," Metals Progress, v 62, p 90, November 3, 1952.
- Gruen, E. J., "Coating Magnesium Alloys," <u>Light Metal Age</u>, March 1944.
- Hanawalt, J. D., Nelson, C. E., and Peloubet, J. A., "Corrosion Studies of Magnesium and Its Alloys," <u>Light Metals</u>, v VIII, p 492, 1945.
- 20. Huber, K., "Anodic Formation of Coatings on Magnesium," Electrochemical Society Journal, v 100, p 376-82, August 8, 1953.
- 21. Keso, Edward E. and Self, Huber, "The Magnesium Industry," Reprint from Economic Geography, A & M Collection.
- 22. Kohl, A. L. and Waterman, H., "A New Surface Treatment for. Magnesium," v 161, p 50-55, The Iron Age, January 22, 1948.
- 23. Loose, W. S. and DeLong, H. K., "Surface Treatment of Magnesium Alloys," Metal Progress, p 899, May 1944.
- 24. Mason, R. B., "A Protective Finish for Magnesium Alloys," The Iron Age, v 157, p 48-52, March 21, 1946.
- 25. McNulty, R. E. and Hanawalt, J. D., "Some Corrosion Characteristics of High Purity Magnesium Alloys," <u>Transactions of</u> the Electrochemical Society, v 81, 1942.
- 26. Pitman-Dunn Laboratories, Frankford Arsenal, Philadelphia, Pennsylvania, "Supplemental Information on the HAE Coating for Magnesium," February 25, 1954.
- 27. Starr, J., "Finishing Magnesium," <u>Metal Finishing</u>, v 50, p 62-4, October 1952.
- Sutton, H. and LeBrocq, L. F., "Protection of Magnesium and its Alloys," Journal of the Institute of Metals, v 57, p 199-227, 1935.
- 29. Tupholme, C. H. S., "Surface Treatment of Magnesium Alloys," Light Metals, v 1, p 129, 1938.
- 30. Wernick, S. and Pinner, R., "Surface Treatment and Finishing of Light Metals," Metal Finishing, v 52, p 5609, March 1954.
- Anon., "Corrosion and Protection of Magnesium Alloys," Light Metals, series of articles, v 2, p 218, 243, 274, 313, 1939.

APPENDIX

Method of Identifying Panels

- 1. Panels are first designated by a Roman Numeral code number. This code number is either I, II, or III, and may be determined from the number of notchs in the upper left hand corner of each panel.
- 2. Panels are further designated by the position of a single hole through the panel. The location of the hole may be determined by the below coordinate system.







With the notch in the upper left hand corner we immediately know that this must be a code I panel.

Further, if a hole were in position (1), this panel would be further designated as a B-5 panel. The complete designation would of course be I-B-5.

Should the hole be in position (2), we would then have panel I-E-2.

Duplicate panels are prepared in all cases, there is no differentiation made between the duplicate panels.

Composition of the HAE Bath for Magnesium

Potassium Hydroxide (KOH) Tech.16 oz.Aluminum Hydroxide $(Al(OH)_3)$ U.S.P.4 oz.Potassium Fluoride (KF) Tech.4.5 oz.Trisodium Phosphate (Na₃PO4:12 H₂O) Tech.4.5 oz.Potassium Manganate (97% K₂MnO₄)2.5 oz.Water-to make.1 gal.

Table 3

Composition of the Dow #12 Caustic Anodize Bath

Sodium Hydroxide (NaOH) Tech.	32	oZ,
Ethlene Glycol (CH2OHCH2OH) U.S.P.	•55	pint
Sodium Oxalate (Na2C204) U.S.P.	•33	oz.
Water -to make-	l	gal.

Table 4

Composition of the Neutralizing Bath

Sodium	Acid Fluoride	(NaHF))	Tech.		6,66	oz.
Sodium	Dichromate (Na	Cr 0	ş	2H O)	Tech.	6.66	oz,
Water	-to i	nake-				1	gal.

Composition of the Dow #14 Anodize Bath

Phenol	(C ₆ H ₅ OH) U.S.P.	1 . 0	02.
Sodium	Hydroxide (NaOH) Tech.	•33	oz,
Sodium	Metaborate (Na ₂ B ₂ O ₄ : 4 H ₂ O) Tech.	32.0	oz.
Sodium	Metasilicate (Na ₂ SiO ₃ : 9H ₂ O) Tech.	9.0	oz.
Water	-to make-	1.0	gal.

Table 6

Composition of Dow #17 Anodize Bath

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Ammonium Acid Fluorid	le (NH4HF2) Tech.	32 0	z.
Sodium Dichromate (Na	$1_2 \operatorname{Cr}_2 \operatorname{O}_7$: $2 \operatorname{H}_2 \operatorname{O}$) Tech.	13 . 3 o	z.
Phosphoric Acid (85%	H ₃ PO ₄) Reagent	11.5 Fl.o	z.
Water	-to make-	l g	al.

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Diagramatic View of Process Arrangement



Operation Treatment Time Temperature (Minutes) Alkaline Cleaning 10 190-212 Cold Water Rinse 1 200 Chromic Acid Dip . . Cold Water Rinse Nitric Acid Pickle 2 70 Cold Water Rinse Scrubbing Air Dry Room Temp 6.25-50 165-175 Anodize Cold Water Rinse 5 70-90 Neutralizing Cold Water Rinse Air Dry Room Temp

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Flow Chart for Dow #12 Process

Flow Chart for Dow #14 Anodize for Magnesium

Operation	Treatment Time (Minutes)	Temperature (°F.)
Alkaline Cleaning	10	190-212
Cold Water Rinse		
Chromic Acid Dip	l	. 200
Cold Water Rinse		
Nitric Acid Pickle	2	70-85
Cold Water Rinse		
Scrubbing		
Air Dry Room Temp.		
Anodizing	5-40	85-90
Cold Water Rinse		
Air Dry Room Temp.		

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Operation Treatment Time Temperature (°F) (Minutes) 10 190-212 Alkaline Cleaning Cold Water Rinse 1 200 Chromie Acid Dip Cold Water Rinse Nitric Acid Pickle 2 70-85 Cold Water Rinse Scrubbing Air Dry Room Temp. Anodizing 1.25-80 160-180 Cold Water Rinse Air Dry Room Temp.

Flow Chart for Dow #17 Anodize for Magnesium

Flow Chart for the HAE Coating Process



Record of Treatments

Dow #12 Anodize Process

Panel I. D.	%Rec. C. D.	%Rec. Time	Actual C. D.	Actual Time	Ammeter Reading	Start	Stop	Date
III-A-4/6	200	200	40	50	6,66	7:06	7:56	3/13
III-A-10/1	L200	150	40	37.5	6.66	6:27	7:04+	3/13
II-A-10	200	100	40	25	6.66	4:31	4:56	3/13
I-A-8	200	50	40	12.5	6.66	4:07	4:19+	3/12
I-A-7	200	25	40	6,25	6,66	3:59	4:05+	3/12
II-A-7	150	200	30	50	5.0	3:00	3: 50	3/12
II-A-6	150	150	30	37,5	5.0	2:12	2:49+	3/12
I-A-6	150	100	3 0	25	5.0	1:45	2:10	3/12
II -A- 5	150	50	30	12.5	5.0	1:22	1:34+	3/12
I-A-5	150	25	30	6.25	5.0	1:12	1:18+	3/12
I-A-4	100	200	20	50	3.33	9:47	10:37	3/11
II-A-4	100	150	20	37.5	3.33	9:05	9:42+	3/11
II-A-3	100	100	20	25	3.33	8:34	8:59	3/11
II-A-2	100	50	20	12,5	3.33	8:12	8:24+	3/11
I-A-3	100	25	20	6,25	3.33	5:52	5:58+	3/11
I-A-2	50	200	10	50	1 . 66	4:27	5:17	3/11
I-A-11	50	150	10	37,5	1 . 66	3:48	4:25+	3/11
II-A-11	50	100	10	25	1.66	3:07	3:32	3/11
II-A-l	50	50	10	12.5	1 . 66	10:43	10:55+	3/10
I-A-l	50	25	10	6.25	1.66	10:31	10:37+	3/10
III - A - 1/3	25	200	5	50	•83	5:38	4:28	3/13
I-A-10	25	150	5	37.5	. 83	2:55	3:32+	3/13
II-A-9	25	100	5	25	. 83	2 :2 7	2:52	3/13
I-A-9	25	50	5	12.5	<mark>،</mark> 83	2:13	2:25+	3/13
II-A-8	25	25	5	6.25	.83	4:24	4:30+	3/13

(C.D) Current density measured in Amperes/Square Foot. Time measured in minutes.

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Record of Treatments

Panel	Rec.	%Rec.	Actual	Actual	Ammeter	Start	\mathtt{Stop}	Date
I.D.	C.D.	Time	C.D.	Time	Reading	and the second and sold in the second		
	000	200	70	10	E	1.20	9.10	1/70
1-1-4	200	200	30	40	5 E	12.45	2:10 3.75	4/17
T T O	200	100	30	30	ย ต	10,15	10.2E	4/17
1	200	T00	30 70	20	ย ต	10 00	10,10	4/17
1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-	200 200	90 95	30 70	TO	5			$\frac{4}{17}$
	200	25	30	5	5	11:55	12:00	4/17
I-=Ei5	150 	200	22.5	40	3.75	2:25	3:05	4/17
I-E-6	150	150	22,5	30	3.75	4: 50	5:20	4/17
I-E-7	150	100	22.5	20	3,75	5:25	5:45	4/17
I-E-8	150	50	22.5	10	3,75	3:10	3:20	4/17
I-E-9	150	25	22.5	5	3,75	5:47	5:52	4/17
I-D-10	100	200	15	40	2.5	5 : 50	6:30	4/14
I-D- 9	100	150	15	3 0	2.5	5:15	5:45	4/15
I D-6	100	100	15	20	2.5	3:12	3:32	4/15
I-D-7	100	50	15	10	2.5	2:18	2:28	4/15
I-D-8	100	25	15	5	2,5	1: 50	1:55	4/15
I-D-11	50	200	7.5	40	1.25	2:30	3:10	4/16
II-D-1	50	150	7.5	30	1.25	3:15	3:45	4/16
II-D-2	50	100	7,5	20	1.25	4:13	4:33	4/16
II-D-3	50	50	7,5	10	1,25	8:55	9:05	4/16
II-D-4	50	25	7,5	5	1.25	9:10	9:15	4/16
II-D-5	25	200	3.75	40	₀ 625	9:18	9:58	4/16
II-D-6	25	150	3.75	3 0	. 625	10:00	10:30	4/16
II-D-7	25	100	3.75	20	. 625	10 :3 5	10:55	4/16
II-D-8	25	50	3.75	10	. 625	10:58	11:08	$\frac{1}{4}$
II -D -9	25	25	3,75	5	.625	11:30	11:35	4/17
II-D-10	480	20.8	72	4.16	12	11:40	11:44+	4/17

(C.D.) Current Density measured in Amperes/Square Foot. Time measured in minutes.

Record of Treatments

Dow #17 Anodize for Magnesium

Panel I. D.	%Rec. C.D.	%Rec. Time	Actual C.D.	Actual Time	Ammeter Reading	Start	Stop Date
II-B-10	200	200	100	10	16.65	4:05	4:15 3/15
I-B-11	200	150	100	7.5	16,65	4:17	4:24+3/15
II-B-11	200	100	100	5	16 . 65	4:25	4:30 3/15
III-B-1/2	200	50	1.00	2.5	16 . 65	4:33	4:35+3/15
III-B-3/4	200	25	100	1.25	16,65	4:48	4:49+3/15
I-B-1	150	200	75	13,35	12,50	6:59	7:12+3/14
II-B-1	150	150	75	10.	12,50	3:52	4:02 3/14
II-B-2	150	100	75	6,67	12,50	7:18	7:24+ 3/14
I-B -2	150	50	75	3,33	12.50	7:28	7:31+ 3/14
I-B-3	150	25	75	1.67	12,50	7 :3 5	7:36+ 3/14
II-B-3	100	200	50	20	8.32	7:42	8:02 3/14
I-B-4	100	150	50	15	8 :32	8:03	8:18 3/14
II-B-4	100	100	50	10	8.32	8:24	8:34 3/14
I-C-5	100	50	50	5	8,32	8:48	8:53 3/14
I ∞ B∞6	100	25	50	2.5	8.32	9:05	9:07+ 3/14
I-B-9	50	200	25	40	4.16	2:17	2:57 3/15
II-B-9	50	150	25	30	4,16	2:59	3:29 3/15
I-B-10	50	100	25	20	4.16	3:31	3:51 3/15
I-B-8	50	50	25	10	4.16	11:41	11:51 3/14
II-B-8	50	25	25	5	4.16	11:54	11:59 3/14
II - B-6	25	200	12.5	80	2,08	9:12	10:32 3/14
III-B-6/7	25	150	12.5	60	2,08	7:10	8:10 3/15
III-B-10/	11 25	100	12.5	40	2,08	8:20	9:00 3/15
II-B-7	25	50	12.5	20	2,08	11:19	11:39 3/14
I=B=7	25	25	12.5	10	2.08	11:05	11:15 3/14

(C.D.) Current Density measured in Amperes/Square Foot. Time measured in minutes.

Record of Treatments

HAE Coating for Magnesium

Panel	%Rec.	%Rec.	Actual	Actual	Ammeter	Start	\mathtt{Stop}	Date
I. D.	C. D.	Time	C. D.	Time	Reading	magned (Salar Capital (Salar Salar)	ou with the second second	-
т ټ өж	200	150	30	1 2 5	Ę	1.00	3.15	ALA
	200	150	30	135	5	1;00 7.13	0,20	
	200	100	30	700	5	3.20	4.50	A/A
I-F-3	200	50	30	45	5	11.00	11.45	π/π Δ/Δ
TIFF	200	50	30	45	5	4.50	5.35	$\frac{1}{4}$
TTaCas	200	25	30	22.5	5	11,35	11.57+	3/30
T_D_2*	150	166	22.5	150	3.75	1.45	4.15	4/5
I-D-1	150	150	22.5	135	3,75	9.35	11:50	4/5
II-F-4	150	100	22.5	90	3,75	10:30	12:00	4/4
II-F-3	150	50	22.5	45	3,75	9:42	10:27	$\frac{1}{4}/4$
II C 10	150	25	22.5	22.5	3.75	4:50	5:12+	3/30
I-C-10	100	200	15	180	2.5	9:00	12:00	3/28
I-C-11	100	150	15	135	2.5	8:50	11:05	3/29
I -C- 9	100	100	15	90	2,5	7:20	8:50	3/28
I-C-8	1.00	50	15	45	2.5	6:30	7:15	3/28
I-C-7	100	25	15	22.5	2.5	6:05	6:27+	3/28
I-C-2	50	200	7.5	180	1.25	12:55	3:55	3/26
II-C-3	50	150	7.5	135	1,25	6:45	9:00	3/29
II-C-2	50	100	7.5	90	1.25	3:55	5:25	3/29
II-C-l	50	50	7.5	45	1,25	11:10	11:55	3/28
II-C-4	50	25	7.5	22.5	1.25	9:05	9:27+	3/29
II-C-9	25	200	3.75	180	.625	1:10	4:10	3/30
II⊷C⇔7	25	150	3.75	135	. 625	9:15	11:30	3/30
I-F-1	25	100	3. 75	90	.625	9:12	10:42	4/4
II-C-6	25	50	3.75	45	。 625	10:41	11:26	3/29
II-C-5	25	25	3,75	22.5	.625	9:37	9:59+	3/29

*Panels were withdrawn from the bath in less than scheduled time as the coating began to fail with further increases in potential.

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(C. D.) Current density measured in Amperes/Square Foot. Time measured in minutes.

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Panels Treated By the Dow # 12 Process

For Weathering on the Gulf

Coast of Texas

Panel Identification	Current Density Amperes/Sq. Ft.	Time Minutes	AppearanceRemarks
III-A-4	40	50	Lt. gray green
III-A-lO	40	37.5	Lt. gray green, some spots on one side
II-A-10	40	25	Lt. gray green
I-A-8	4 0	12.5	Soft gray
I-A-7	40	6,25	Lt. gray
II-A-7	30	50	Med. gray green
II- A- 6	30	37.5	Med. gray with gray green about edges
I-A-6	30	25	Lt. gray green
II-A-5	30	12.5	Soft gray, some light streaking
I-A-5	30	6.25	Silvery white, one edge lightly pitted
I-A-4	20	50	Smooth black
II-A-4	20	37.5	Smooth black
II-A-3	20	25	Med. gray
II-A-2	20	12.5	Very light gray
I-A-3	20	6.25	Lt. gray
I-A-2	10	50	Med. dark gray green
I-A-11	10	₂ 37 . 5	Med. gray, streaked on one side
II-A-11	10	25	Soft gray
II-A-l	lo	12.5	Very light gray
I-A-1	10	6.25	Uneven Lt. gray, many fine pits
III-A-3	5	50	Very thin gray film
I-A-10	5	37.5	Silvery white
II-A-9	5	25	Silvery white
I A. 9	5	12.5	Metallic colored, water streaked
II-A-8	5	6,25	Bare metal

Panels Treated By the Dow #14 Process

For Weathering on the Gulf

Coast of Texas

Panel	Current Density	Time	
Identification	Amperes/Sq. Ft.	Minutes	Appearance-Remarks
I-E-4	30	40	Light gray-rough
I-E-3	30	30	Light gray-rough
I-E-2	30	20	Light gray-rough
I-E-1	30	10	Light gray-slick
II-D-11	30	5	Light gray-slick
I-E-5	22.5	40	Light gray-rough
I-E-6	22.5	30	Light gray-rough
I-E-7	22.5	20	light gray-rough
I-E-8	22.5	10	Light gray-slick
I-E-9	22.5	5	Light gray-slick
I-D-10	15	40	Light gray-rough
I-D-9	15	30	Light gray-rough
I-D-6	15	20	Light gray-slick
I-D-7	15	10	Light gray-slick
I-D-8	15	5	Light gray-slick
I-D-11	7.5	40	Light gray-slick
II-D-1	7.5	30	Light gray-slick
11-D-2	7.5	20	light gray-slick
II-D-3	7.5	10	Light gray-very thin
II-D-4	7,5	5	Lt. gray-10% exposed
II-D-5	3.75	40	Light gray-slick
II-D-6	3.75	30	Light gray-slick
II-D7	3,75	20	Lt. gray-5% exposed
II-D-8	3° 22	10	Lt. gray-80% exposed
II - D-9	3.75	5	Lt. gray-95% exposed
II-D-10	72	4.16	Light gray-slick

Panels Treated By the Dow #17 Process

For Weathering on the Gulf

Coast of Texas

Panel	Current Density	Time	
Identification	Amperes/Sq. Ft.	Minutes	Appearance-Remarks
II-B-10	100	10	Dark green
I-B-11	100	7.5	Dark green
II-B-11	100	5	Dark green
III-B-l	100	2,5	Lime green
III-B-4	100	1,25	Chartreuse
I-B1	75	13.35	Dark green
II-B-l	75	10	Dark green
II-B-2	75	6,67	Dark green
I-B-2	75	3.33	Lime green
I-B-3	75	1.67	Pale chartreuse
II-B-3	50	20	Dark green
I-B-4	50	15	Dark green
II-B-4	50	10	Dark green
I-C-5	50	5	Lime green
I-B-6	50	2.5	Pale chartreuse
I-B-9	25	40	Dark green
II-B-9	25	30	Dark green
I-B-10	25	20	Dark green
I-B-8	25	10	Lime green
II-B-8	25	5	Chartreuse
II-B-6	12.5	80	Dark green
III-B-6	12.5	60	Dark green
III-B-11	12.5	40	Dark green
II-B-7	12.5	20	Lime green
I-B-7	12.5	1.0	Chartreuse

Panels Treated By the HAE Process

For Weathering on the

Gulf Coast of Texas

Panel Current Density Time	
Identification Amperes/Sq. Ft. Minutes Appearance-Re	marks
I-F-2 30 135 Dark brown, 1 burned throug	/16 ^w ho le h
II-C-ll 30 135 Dark brown, 3 1/2 sq in exp one edge	holes, osed along
II-F-2 30 90 Med. dark bro	wn
I-F-3 30 45 Med. Dk. brow some light pa	n with tchs
II-F-l 30 45 Med. Dk. brow some light pa	n with tchs
II-C-8 30 22.5 Splattered Br	. over tan
I-D-2 22.5 150 Dark brown	
I-D-1 22.5 135 Dark brown, i slightly atta	ndex edge cked
II-F-4 22,5 90 Dk. Br. few 1	ight spots
II-F-3 22.5 45 Med. Dr. Br. light patchs.	with many
II-C-10 22.5 22.5 Splattered Br	. over tan
I-C-10 15 180 Dark brown	
I-C-11 15 135 Medium dark b	rown
I-C-9 15 90 Lt. Br., with lighter patch	many s
I-C-8 15 45 Tan with spla light brown	ttered
I-C-7 15 22.5 Tan with fain shaded areas	t brown
I-C-2 7.5 180 Med. Br. with lighter patch	many s
II-C-3 7.5 135 Tan with much splattering	Med. Br.
II-C-2 7.5 90 "	
II-C-1 7.5 45 Tan with Med.	Br. shading
II-C-4 7.5 22.5 Light tan	
II-C-9 3.75 180 Tan with scat	tered Br.
TT=C=7 3,75 335 *	ť
$T_{\rm F} = 1$ 3.75 90 ⁸ around	edres
$II_{-}C_{-}6$ 3_75 45 Light ten	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
II-C-5 3.75 22.5 Very light ta	n

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Results of Weathering Dow #12 Coating

Panel Identi fi cati on	Current	Amp-min Treatment	% Failure	Rela	tive
	$Amps/ft^2$	(per ft ²)	Salt Fog	Visual	% Wise
in gas				_	
III-A-6	40	2000	.2	1	1
III-A-11	40	1500	.24	2	2
II- A-1 0	40	1000	.25	5	3
I-A-8	40	500	.43	8	lo
I-A-7	40	250	•47	6	11
II-A-7	30	1500	.31	3	6
II-A-6	30	1125	. 28	9	4
I-A-6	30	750	。 38	13	9
II-A-5	30	375	.50	7	12
I-A-5	30	187.5	。 93	17	17
I-A-4	20	1000	.29	11	5
II-A-4	20	750	.36	15	8
II-A-3	20	500	.33	4	7
II-A-2	20	250	. 57	10	14
I-A-3	20	125	.52	12	13
I-A-2	10	500	. 86	14	16
I-A-11	10	375	3.0	22	18
II-A-11	10	250	。 66	16	15
II-A-l	10	125	3.0	18	19
I-A-1	10	62.5	4.0	19	20
III-A-l	5	250	20.0	20	21
I-A-10	5	187.5	60.0	24	24
II-A-9	5	125	55.0	21	23
I-A-9	5	62.5	50.0	23	22
II-A-8	5	37.5	95.0	25	25

Results of Weathering Dow #14 Coating

_	Current	Amp-min	% Failure	Rela	tive
Panel	Density ₂	Treatment	200 Hours	Stand	aing
Identification	Amps/it	per sq it	Salt Fog	Vlsual	% Wise
I-E-4	30	1200	.20	3	1
I-E-3	30	900	.22	4	3
I-E-2	30	600	。 40	6	6
I-E-1	30	300	.92	9	12
II-D-11	30	150	9.0	15	16
I-E-5	22.5	900	。 35	1	4
I-E-6	22.5	675	.20	2	2
I-E-7	22.5	450	.36	7	5
I-E-8	22.5	225	3.0	13	14
I-E-9	22.5	112,5	7.5	16	15
I-D-10	15	600	•47	5	8
I-D-9	15	450	•58	10	9
I-D-6	15	300	.61	11	10
I-D-7	15	150	35.	20	19
I-D-8	15	75	45.	22	20
I-D-11	7.5	300	.43	12	7
II-D-l	7.5	225	.94	14	13
II-D-2	7.5	150	30.	17	18
II-D-3	7.5	75	45.	19	21
II-D-4	7.5	37.5	55.	21	22
II-D-5	3.75	150	18.	18	17
II-D-6	3.75	112.5	60.	26	24
II-D-7	$3_{\bullet}75$	75.	55,	23	23
II-D-8	3.75	37.5	85.	24	25
II-D-9	3.75	18.75	95.	25	26
II-D-10	72	300	•93	8	11

Results of Weathering Dow #17 Coating

The set of T	Current	Amp-Min	% Failure	Rela	tive
Panel	Density	Treatment	200 Hours	D Can	uing William
Identification	Amps/17	per sd it	Sart rog	VISUAL	% W186
II-B-10	100	1000	. 069	3	3
I-B-11	100	750	.125	9	11
II-B-11	100	500	.069	7	4
III-B-2	100	250	.077	14	5
III-B-3	100	125	.48	25	25
I-B-l	75	1000	.035	1	1
II-B-1	75	750	.125	13	12
II-B-2	75	500	.098	15	8
I-B-2	75	250	.18	22	19
I-B=3	75	125	.28	20	24
II-B3	50	1000	.091	13	6
I-B-4	50	750	.125	18	13
II-B-4	50	500	。 056	4	2
I-C-5	50	250	.24	19	22
I-B-6	50	125	.195	21	20
I-B-9	25	1000	.125	6	14
II-B-9	25	750	.14	11	16
I-B-10	25 -	500	. 175	10	18
I-B-8	25	250	.12	17	9
II-B-8	25	125	.23	24	21
II-B-6	12.5	1000	.091	5	7
III-B-7	12.5	750	,125	8	15
III-B-lO	12.5	500	.14	16	17
II-B-7	12.5	250	.12	18	10
I-B-7	12.5	125	.27	23	23

Results of Weathering HAE Coating

PanelDensitIdentificationAmps/fI-F-230II-C-1130	y 1reathe <u>t</u> ² <u>per sq</u> 4050 4050 2700	nt 200 hour ft Salt Fog .035 .042	-s Sc <u>Visua</u> 4 7	anding <u>1 % Wise</u> 3
I-F-2 30 II-C-11 30	4050 4050 2700	.035 .042	4. 7	,
I-F-2 30 II-C-11 30	4050 4050 2700	.035 .042	4 7	3
II-C-11 30	4050 2700	,042	7	
	2 700		•	4
II-F-2 30		2021	1	1
I -F-3 30	1350	.16	10	10
II-C-8 30	675	。 36	14	14
I-D-2 22.5	3375	.091	8	8
I-D-1 22.5	3037	.069	6	7
II-F-4 22.5	2025	.091	9	9
II-F-3 22.5	1012.5	.26	11	11
II-C-10 22.5	506.2	. 68	20	20
I -C-1 0 15	2700	.021	2	2
I-C-11 15	2025	.042	5	5
I-C-9 15	1350	.049	3	6
I-C-8 15	675	. 35	17	13
I-C-7 15	337. 5	.67	19	19
I-C-2 7.5	1350	.26	12	12
II-C-3 7.5	1012.5	. 62	15	18
II-C-2 7.5	675	. 49	18	15
II-C-1 7.5	3 37 . 5	.89	21	22
II-C-4 7.5	168.7	8.	25	25
II-C-9 3.7	5 675	•49	13	16
II-C7 3.7	5 506.2	•54	16	17
I-F-1 3.7	5 337 . 5	.81	22	21
II-C-6 3.7	5 168.7	3.	24	23
II-C-5 3.7	5 84.3	5.	23	24



Ammeter Readings (Amperes)

Voltage Rise During an

HAE Treatment Cycle

Process: HAE Current Density: 50 amperes/square foot Total treatment time: 45 minutes

Minutes	<u>Volts (ac</u>)
。 5	59.0
l	60,0
2	63,5
3	66.0
4.	66.5
5	67.0
6	67.5
7	68.0
8	68,5
9	68.5
10	68,5
15	69.5
20	70.5
30	71.5
45	75,5

Figure 12 Voltage Rise During a HAE Treatment Cycle



Voltage Rise During a Dow #17 Anodize Treatment Cycle

Process: Dow #17 Anodize for Magnesium Current Density: 12.5 Amperes/Square foot Treatment time: 60 minutes

Minutes	Volts (ac)
•5	46 •0
1	50.0
2	55.0
- 3	62.5
4	65,5
5	67.5
6	69 。 0
8	71.0
10	72,6
15	76 . 0
20	78.0
30	82.0
4 O	85,5
50	87.0
60	87 . 5



Appearance of Coated Panels After 200 Hours in Salt Fog



Power Consumed During a Dow #17 Treatment Cycle:

From Figure 13 which is a plot of the data from Table 20, a summation of the area under the curve will determine the power consumed.

Area I = 6 min x 59.3 volts = 355.8 volt-min. II = 6 x 72.0 = 432.0 III = 18 x 78.5 = 1413. IV = 30 x 86.0 = 2580. 4780.0 volt-min. <u>Kw-hr.= volt-min x amps/sq. ft.</u> = 4780×12.5 = .997 sq.ft. 1000 x 60 = .997 .997 Kw-hr/sq.ft. represents the power to apply a 750 ampere-minute treatment. This is 150% of the maximum recommended treatment. Prorated to 100% of the maximum recommended treatment:

<u>.997 Kw-Hr./sq.ft.</u> = <u>0.665 Kw-hr./sq.ft.</u> 1.5

Power Consumed During an HAE Treatment Cycle:

From Figure 12 which is a plot of the data from Table 19, a summation of the area under the curve will determine the power consumed.

Area	I II	11 11	5 5	min.	x 63.5 x 68.3	volts	125 25	317.5 341.5	volt-mi	ln
	III		35	-	x 72.0)		$\frac{2420.0}{3079.0}$	volt-mi	Ln
Kw-Hr sq ft		volt	-min 1000	. x am x 60	ps/sq_	<u>ft</u> =	307 100	79 x 50 00 x 60	= 2.51	Kw-hr sq ft
-	** 1	1	~							~ _

2.51 Kw-hr/sq ft represents the power to apply a 2250 ampere-minute treatment. This is 166% of the maximum recommended treatment.

Prorated to 100% of the maximum recommended treatment:

Area of Panel:

$$\frac{2.75 \text{ in x 4 in}}{144 \text{ sq in/sq ft}} = .0764 \text{ sq ft/ per surface}$$
Current Density:
Ammeter Reading/Effective Area
Effective Area = Panel area + Electrode area

$$\frac{2 \times 2.75^{n} \times 4^{n} + 3/8^{n} \times 4 \times 1.5^{n} - (3/8)^{2} \times 2}{144} =$$

$$\frac{1665 \text{ sq ft per panel with electrode attached}}{144}$$

Ampere-Minutes Treatment:

Ammeter reading x Minutes = Current density x time Effective area

Percent Area Failure:

Number of pits x pit area x 100 = % Failure panel area

$$\frac{\text{Pi x (1/32)}^2 \times \text{N x 100}}{4 \times 144}$$

 $= 6.97 \text{ N x } 10^{-3} \%$

.0764

For panel I=F=2: N = 5 % Failure = 5 x 6.97 x 10^{-3} = .035%

VITA

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