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Electro-Galvanizing from a Purified Ammonium Sulphate Leach of Anaconda Zinc Calcine

Phillip J. Mischkot Jr.

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ELECTRO-GALVANIZING FROM A PURIFIED AMMONIUM
SULPHATE LEACH OF ANACONDA ZINC CALCINE

By

Philip J. Mischkot, Jr.

A Thesis

Submitted to the Department of Metallurgy
in Partial Fulfillment
of the Requirements for the
Degree of Bachelor of Science
in Metallurgical Engineering

MONTANA SCHOOL OF MINES

BUTTE, MONTANA

May 12, 1951

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FOREWORD

"One of the most persistent problems which confront the worker in iron and steel is the prevention of corrosion. We cannot rid ourselves of the agents which affect corrosion of iron without at the same time ridding ourselves of the agents which are essential to life itself."¹

Alfred Sang

"THE IRON AGE"

INTRODUCTION

Ever since the introduction of metal products into our civilization, especially those of iron and steel, man has been confronted with the problems of corrosion prevention. As the agents which are conducive to corrosion can not be completely eliminated, it has been necessary for man to develop processes and materials that will retard the degeneration of the metals that are so important to our industrialized world. Even though our technical efforts in combating corrosion have been persistent and successful in most instances, the damage to iron and steel products alone amounts to approximately four billion dollars annually.² This value does not include the losses of life and limb that are the direct results of corrosive damage.

Iron and steel are protected in order to prevent decomposition by two common ways, namely metallic and non-metallic methods.³ The non-metallic method consists of covering the metal with paint or varnish which are

¹•Flanders, W. T., Galvanizing and Tinning (New York: 1916), p. 9.

²•C. E. Heussner, "Protection of Base Metals by Use of Metallic Coatings." Symposium on Protecting Metals Against Corrosion, 1938.

³•Flanders, p. 9.

essentially composed of organic constituents. This coating prevents corrosion by keeping the atmosphere from coming into contact with the base metal. As long as the film is not broken, i.e., it remains airtight, the metal surface will be protected. The metallic method consists in coating the metal surface with another metal by one of several processes. It is with this method that we are concerned in this problem.

Of all the metallic coatings used today for the protection of iron and steel, such as, zinc, chromium, nickel, lead, tin, cadmium, and copper; the zinc coating is one of the oldest, the most economical, and by far the most widely used. The zinc coating itself does not resist corrosion, in fact, its usefulness in corrosion prevention of the base metal is dependent upon its corroding.

Two dissimilar metals form a galvanic couple when exposed to the atmosphere, which is essentially an electrolyte. In the case of galvanized iron or steel, the zinc, which is more electropositive than the base metal, corrodes while the electronegative base metal is not affected. The galvanic action is very slight under ordinary atmospheric conditions and a light zinc coating will last for a long time. Of course, if more

corrosive conditions are encountered, the life of the coating is decreased accordingly. The thickness of the coating does not affect the galvanic action and the only advantage of thicker coatings is that the base metal will be protected for a longer period of time. The composition of the zinc coating and the base metal affects the rate of corrosion to a great extent. This fact is ably expressed by Mr. Alfred Sang in "The Iron Age".

"Iron rusts less easily than does steel; this is perhaps due to steel being a very composite material. In the iron, which forms the bulk of its composition, are dissolved or immersed a great variety of other substances; some of these are simple, such as graphite, silicon and manganese, and others are compound, such as carbides, sulfides, phosphides, and silicides. The carbon compounds are very numerous and diversified, being due to different heat treatments; the best known are cementite, pearlite and martensite. Just as variety is to some people the spice of living, so is heterogeneous composition the spice of rusting, in the present instance at any rate. Nor is this by any means a solitary instance; it is a well-known fact that chemically pure zinc is dissolved very slowly by certain acids, whereas the commercial product, especially if it be high in iron, is rapidly dissolved."⁴

⁴•Flanders, p. 10.

Galvanizing Methods:

There are four commercial methods of applying zinc coatings to iron and steel. They are as follows:⁵

1. Hot dipping
2. Electrogalvanizing
3. Sherardizing
4. Spray coating

Hot dipping is by far the oldest and most widely used method of galvanizing. It is not the purpose of this work to discuss the hot dipping methods, its merits or its shortcomings, but it is necessary to point out the defects in the products of this method in order to show the need and the further development of electrogalvanizing.

The demand of industry for a galvanized product which has a pure, ductile, fine-grained, adherent zinc coat forced the development of electrogalvanizing. Probably the most important reason for this development is the need for a method to increase the thickness of the coating and at the same time retain a resistance to cracking when the product is deformed. It is well known that the thickness of coating in the hot dip process is limited. Furthermore, because of

⁵ American Society for Metals, Metals Handbook (Cleveland, 1948) pp. 712-714.

the formation of a zinc compound instead of pure zinc coating, the protective layer will crack when deformed. Sherardizing and spray coating are methods of galvanizing which are confined to special products.

Electro galvanizing:

It is not the purpose of this paper nor is there a need to discuss the fundamental principles of chemistry, electricity, and electrochemistry. These principles are adequately covered in several good text books on the above subjects.^{6,7,8} However, it should be noted that these principles are very important inasmuch as they determine the efficiency of electro-deposition and therefore the cost of depositing a definite amount of metal.

The characteristics of a metal coating, such as hardness, softness, smoothness, brightness, and adherence are determined by the physical structure of the deposited metal. It is commonly known that the process

⁶•H. Jermain Creighton, Principles and Applications of Electrochemistry, Vol. I--Principles (New York, 1943).

⁷•W. A. Koehler, Principles and Applications of Electrochemistry, Vol. II--Applications (New York, 1944).

⁸•William Blum, Principles of Electroplating and Electroforming, (New York, 1930).

of electrodeposition depends upon the formation of crystals; therefore, it is essential that the several factors or a combination of these factors which influence crystal formation by electrodeposition be taken into consideration. These factors are as follows:⁹

1. Current density
2. Concentration and agitation
3. Temperature
4. Conductivity
5. Metal ion concentration
6. Hydrogen ion concentration
7. Addition agents
8. Structure of the base metal
9. Metal distribution and throwing power

Although all of these are very important either singularly or in combination with each other, only current density, concentration, agitation, temperature, and addition agents were considered in this work.

Current density is important inasmuch as it controls the time during which a given weight of metal can be deposited. However, the effect of current density upon the character of the deposited metal is of

⁹Blum, pp. 86--119.

primary importance. Generally, a higher current density gives a finer crystal structure even though this may seem contrary to practical experience. Rough, "burnt", and "treed" deposits are usually associated with high current density, but these deposits, even though their outward appearances do not show it, have a fine crystal structure. Of course, if the current density is excessively high in relation to other conditions, such as concentration and agitation, a poor deposit will result.

The greater the concentration of the metal ion in the solution, the less chance there will be of local cathodic impoverishment. When dilute solutions are desirable, cathodic impoverishment can be eliminated to a large extent by agitation. Another advantage of agitation, especially in zinc deposition, is the removal of gas bubbles from the cathode. If the agitation is too vigorous, the metal will become "streaked" or the cell sediment will become suspended in the solution and interfere with the galvanizing operation. The effects of temperature upon the character of the deposit are indirect. Other factors, such as concentration, conductivity, and pH, are affected directly by temperature and these in turn

influence the type of deposit. In general, an increasing solution temperature produces coarser crystals.

"Addition reagents"* , organic or inorganic, are added to the electrolyte to form colloids which will migrate to the cathode and reduce the size of the crystals. In many cases, these reagents have a tendency to reduce "treeing".

In order to produce an adherent metal coating when galvanizing iron and steel, the base metal surface must be scrupulously clean. This is especially true when electrogalvanizing. Any grease, oxides, or oxide inclusion must be removed, or in the case of oxide inclusions some provisions must be made to nullify their effects on the surface of the base metal. Greases are usually removed with organic solvents or hot alkali solutions, whereas oxide coatings are eliminated by pickling the surface to be plated in an acid solution. The most effective method of eliminating the effects of oxide inclusions is by electrolyzing the object in a molten alkaline salt such as sodium hydroxide. The oxides of iron are reduced by the

* "Addition reagents" are arbitrarily defined by Blum and Hogaboom as: "Those substances which do not produce any marked effect upon the conductivity, metal ion concentration, or hydrogen ion concentration of solutions, but which do influence the structure of the deposits."

action of the sodium to sponge iron which is subsequently removed by reversing the polarity of the object in used electrolyte.¹⁰

There are only two types of solutions used in commercial electrogalvanizing. These are the zinc acid sulfate and the zinc alkaline cyanide baths. The cyanide type bath is used extensively to produce a mirror finish on odd shaped objects. The acid sulphate bath, which is used exclusively in the electro-winning of zinc from purified leach solution, is utilized in the electrogalvanizing of iron wire and sheet by essentially the same procedure.¹¹

With the advent of leaching zinc calcine with ammoniacal solutions and the subsequent successful recovery of pure zinc from the purified solutions by electrolysis, it seems logical that ammoniacal zinc leach solutions could be used in electrogalvanizing with many added advantages. Of these advantages, the more important are as follows:

1. The elimination of expensive steps in electrogalvanizing processes, such as the production of pure zinc and its subsequent melting and casting into usable forms.

¹⁰. American Zinc Institute, Journal of the American Zinc Institute (St. Louis, Mo.: 1937) p. 45.

¹¹. Ibid., pp. 42--56.

2. Ammoniacal solutions in which the metal ion is combined as a complex ion produce finer-grained deposits than acid sulphate solutions when they are electrolyzed.

3. The leach solutions are regenerated.

4. Ammoniacal leach solutions are easily purified and filtered.

It was with these factors in mind that the following experimental work was carried out by the author.

PRELIMINARY PROCEDURE

Leaching and Purification:

In an attempt to reproduce conditions that could be used in an industrial process of this type, the zinc ammonium sulphate solution was obtained by actually leaching the zinc calcine with fresh leach solution. The zinc calcine conformed to the following analysis.*

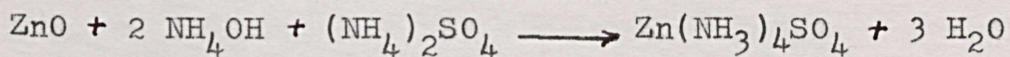
Zinc	56.7%
Copper	1.04%
Cadmium	0.12%
Cobalt	0.90%
Manganese	0.40%
Lead	3.30%
Arsenic	0.18%
Antimony	0.22%
Iron	6.50%
Sulphur	3.40%

The oversize was screened out and only the minus 8-mesh material used as leach feed.

As the primary aim of this work is electro-galvanizing, the chemistry of the leaching and purification will not be discussed in detail. The principal reaction that takes place during the leaching

* C. J. Bartzen, Montana Bureau of Mines, Butte, Mont.

process is as follows:



The solutions used for leaching were prepared on the basis of this equation. From this equation, it can be readily seen that half of the ammonia comes from the ammonium hydroxide and half from the ammonium sulphate. From this point on, the concentration of the leaching solutions will be expressed by per cent total ammonia by weight. The ammonia concentration of the leaching solutions was regulated by additions of water.

The results obtained previous to this work by several investigators^{12,13,14} of the ammoniacal leaching of zinc calcine were correlated and their optimum conditions were utilized. These conditions are as follows:

1. NH_3 10%
2. Leaching time one hour
3. Temperature 25 - 30°C

¹²J. G. Grunenfelder, Ammoniacal Carbonate Leach of Anaconda Zinc Calcine and Recovery of the Zinc by Electrolysis (Butte, Montana, 1933).

¹³Arthur Jewet Bovee, Jr., Ammoniacal Sulphate Leach of Anaconda Zinc Calcine and Recovery of the Zinc by Electrolysis (Butte, Montana, 1934).

¹⁴Allison Richard Dyer, The Electro-winning of Zinc from Anaconda Zinc Calcine Leached with Ammonium Sulphate Solutions (Butte, Montana, 1935).

In order to obtain a saturated neutral solution, a 20 per cent excess of calcine over the calculated amount required to neutralize a measured quantity of pure leach solution was used.

The leach was carried out under the above conditions in a closed bottle which was agitated on motor-driven rolls. Upon completion of the leaching cycle, the solution was immediately filtered. The precipitation of basic ammonia salts during the leaching and also during the subsequent purification of the solution presented many difficulties. A solution of high zinc concentration was not obtained because of this precipitation. Of the several leaching and purifying runs, the highest zinc concentration realized was 90 grams per liter. This concentration was further lowered by the continual precipitation of basic sulphate while the solution was standing.

The leaching conditions were arbitrarily changed in an attempt to eliminate the precipitation of these basic salts. They are as follows:

1. NH_3 15%
2. Leaching time three hours
3. Temperature 25 - 30°C

The results of leaching under these conditions were

excellent. There was no precipitation, and solutions containing from 127.5 to 133.7 grams of zinc per liter were obtained.

Purification of the filtered neutral solution was accomplished by adding zinc dust and agitating the solution in closed bottles for a period of three hours. Although 3 grams of zinc per liter of solution is usually sufficient to remove all the impurities, it was found necessary to use twice this amount because of the abnormally high copper content of the calcine. The purified solution was filtered in the presence of excess zinc dust with a laboratory vacuum filter. Zinc content of all solutions was determined by the ferrocyanide method.¹⁵

¹⁵•Edwin G. Koch, Laboratory Manual for Elementary Quantitative Analysis (Butte, Montana, 1947), p. 128.

EXPERIMENTAL PROCEDURE

The theory and the cell reactions of the electrolysis of ammonium zinc sulphate solutions have been adequately discussed by Bovee and Dyer in previous investigations.^{16,17}

As the preparation of the surface to be galvanized is the first physical operation necessary to obtain an adherent coating, the procedures used will be discussed at this time. The two metal cleaning steps that are usually carried out are degreasing and pickling. However, the black sheet iron used in all experiments was annealed after rolling; therefore, there was no grease present on its surfaces. The sheet was pickled in a seven per cent hydrochloric acid solution¹⁸ until the oxide film was removed. The Armco wire was cleaned mechanically with emery cloth.

In order to determine the conditions which would give the best deposit, various experiments were carried out using different current densities and zinc concen-

¹⁶.Bovee, pp. 11--12.

¹⁷.Dyer, p. 20.

¹⁸.Blum, p. 100.

trations. A "Seal of Quality"* of 1 oz per ft² was chosen as the weight of zinc to be deposited. By simple calculation, the weight necessary to produce this "Seal of Quality" on smaller cathodes was found. The time required to deposit this amount of zinc at various current densities and at a current efficiency of 90 per cent was determined from Faraday's law. A coulometer was used to find the actual current from which the efficiency of deposition could be determined. The pregnant solution was diluted with fresh leach solution in the proper ratios to obtain the desired concentrations and thereby maintain the total NH₃ content constant.

Equipment:

Cell: 400 ml beaker equipped with an electrode holder

Agitation: Motor-driven glass rod stirrer

Cell feed: 50 ml burette

Cell discharge: Continuous siphon

Power: 40 v motor generator set

Anodes: Two 2 $\frac{1}{4}$ in. by 3/4 in. 18 - 8 stainless

*Zinc coatings are usually measured in ounces per square foot of surface for most products except sheet. The weight of coating on sheet is stated in ounces per square foot of sheet. Since the sheet is coated on both sides, this weight is twice the average weight of coating per square foot of surface.¹⁹

¹⁹. American Society for Metals, p. 712.

steel sheets. Sides of anodes away from cathode were blanked off with parafin.

Cathode: $2\frac{1}{4}$ in. by $3/4$ in. black sheet iron

These conditions were kept constant:

Solution volume: 350 ml

Cathode and anode spacing: $3/4$ in.

Cathode to anode area (ratio): 2.3 to 1

Total area of cathode submerged: 3 in.²

Cell temperature: 25 to 30°C

Addition agent: one drop of animal glue

Experiment No. 1:

Time: 77.5 min.

Current: 0.416 amp

Voltage: 2.8 v

Current Density: 20 amp/ft²

Zinc. Conc.: 15 g/l

Current Efficiency: 89.5%*

Type of Deposit: Coarse-grained, dirty, streaked, excessively "treed".

* All current efficiencies were based on the total weight of zinc deposited which included any beads or trees.

Experiment No. 2:

Time: 155 min

Current: 0.208 amp

Voltage: 2.4 v

Current Density: 10 amp/ft²

Zinc Conc.: 30 g/l

Current Efficiency: 91.0%

Type of deposit: Medium-grained, slightly streaked, deposit. Very little "treeing".

Experiment No. 3:

Time: 77.5 min

Current: 0.416 amp

Voltage: 2.7 v

Current Density: 20 amp/ft²

Zinc Conc.: 30 g/l

Current Efficiency: 89.5%

Type of deposit: Medium-grained deposit with a very pleasing appearance. Little "treeing".

Experiment No. 4:

Time: 52 min

Current: 0.624 amp

Voltage: 3.0 v

Current Density: 3.0 amp/ft²

Zinc. Conc.: 30 g/l

Current Efficiency: 82.3%

Type of deposit: Fine-grained, streaked deposit.
Little "treeing".

Experiment No. 5:

Time: 39 min

Current: 0.834 amp

Voltage: 3.0 v

Current Density: 40 amp/ft²

Zinc Conc.: 30 g/l

Current Efficiency: 83.1%

Type of deposit: Fine-grained, dirty, streaked
deposit. Excessive "treeing".

Experiment No. 6:

Time: 155 min

Current: 0.208 amp

Voltage: 2.42 v

Current Density: 10 amp/ft²

Zinc Conc.: 60 g/l

Current Efficiency: 91.5%

Type of deposit: Fine-grained, dirty, streaked
deposit. No "treeing".

Experiment No. 7:

Time: 77.5 min

Current: 0.416 amp

Voltage: 2.5 v

Current Density: 20 amp/ft²

Zinc Conc.: 60 g/l

Current Efficiency: 92.5%

Type of deposit: Excessively streaked, beaded deposit. Little "treeing".

Experiment No. 8:

Time: 52 min

Current: 0.624 amp

Voltage: 2.65 v

Current Density: 30 amp/ft²

Zinc Conc.: 60 g/l

Current Efficiency: 62.6%

Type of deposit: Excessively streaked, beaded deposit. Moderate "treeing".

Experiment No. 9:

Time: 39 min

Current: 0.834 amp

Voltage: 2.8 v

Current Density: 40 amp/ft²

Zinc Conc.: 60 g/l

Current Efficiency: 90.9%

Type of deposit: Fine-grained, slightly streaked deposit. Excessive "treeing".

Experiment No. 10:

Time: 155 min

Current: 0.208 amp

Voltage: 2.5 v

Current Density: 10 amp/ft²

Zinc Conc.: 90 g/l

Current Efficiency: 82.3%

Type of deposit: Fine-grained, slightly streaked deposit. Excessive "treeing".

Experiment No. 11:

Time: 77.5 min

Current: 0.416 amp

Voltage: 2.6 v

Current Density: 20 amp/ft²

Zinc Conc.: 90 g/l

Current Efficiency: 95.6%

Type of deposit: Fine-grained deposit with a pleasing appearance but slightly streaked. Moderate "treeing".

Experiment No. 12:

Time: 52 min

Current: 0.624 amp

Voltage: 2.9 v

Current Density: 30 amp/ft²

Zinc Conc.: 90 g/l

Current Efficiency: 93.7%

Type of deposit: Fine-grained, dirty, slightly streaked deposit. Excessive "treeing".

Experiment No. 13:

Time: 39 min

Current: 0.834 amp

Voltage: 3.0 v

Current Density: 40 amp/ft²

Zinc Conc: 90 g/l

Current Efficiency: 92.4%

Type of deposit: Fine-grained, streaked deposit. Excessive "treeing".

By far, the best deposit was obtained when using the conditions in experiments No. 3 and No. 11. Although the deposit obtained in No. 11 was finer grained than the deposit in No. 3, the zinc concentration, 90 g/l, is too high to be feasible in a

practical application. If the cell concentration is high, the volumes of spent electrolyte are excessive, and there will be a large circulating load of zinc in the cell discharge. It was felt that by the use of the proper addition agents a finer grained deposit could be produced with the conditions used in experiment No. 3. For these two reasons, the conditions used in experiment No. 3 were chosen for use in future investigations.

In order to obtain a more representative sample of the product of this process, the size of the cathode was increased. At the same time, "treed" deposits became a major problem. For this reason, many experiments were started but not completed after "treeing" started.

Equipment used in all the following experiments:

Cell: Glass laboratory cell equipped with an electrode holder. Size: 9 in. by 5 in. by $3\frac{1}{2}$ in.

Agitation: Circulating solution

Cell feed: 50 ml burette

Cell discharge: Continuous siphon

Power: 40 v motor generator set

Anodes: 18 - 8 stainless steel

Cathode: $6\frac{1}{2}$ in. by 5 in. black sheet iron

These conditions were kept constant in all the following experiments:

Solution volume: 1500 ml

Total area of cathode submerged: 37.4 in.²

Current: 5.2 amp

Voltage: 3.0 to 4.0 v

Current Density: 20 amp/ft²

Cell temperature: 25 to 30°C

Experiment No. 14:

Time: 3 hr 50 min

Seal of Quality: 3 oz/ft²

Cathode and anode spacing: 3/4 in.

Anode size: 3 in. by 5 in.

Cathode to anode submerged area (ratio): 2.3 to 1

Addition agent: 5 drops of animal glue

Current Efficiency: Not measured

Type of deposit: Very fine-grained deposit.

Excessive "treeing" on edges and on the surface especially between the anodes and the cathode.

Conclusion: The anodes were too close to the cathode; therefore, the current was concentrated in the vicinity of the anode. As current also has a tendency to concentrate on the edges, the current from the other sides of the anodes caused the "treeing" on the edges of the cathode.

Experiment No. 15:

Time: 3 hr 50 min

Seal of Quality: 3 oz/ft²

Cathode and anode spacing: 1 3/4 in

Anode size: 3 in. x 5 in.

Cathode to anode submerged area (ratio): 2.3 to 1

Addition agent: 5 drops of animal glue

Current Efficiency: Not measured

Note: All but the sides of the anodes facing the cathode were blanked off with parafin.

Type of deposit: Same as above, except there was less "treeing" on the edges.

Conclusion: The anodes were too close to the cathode.

Experiment No. 16:

Same conditions as were used in experiment No. 15, except tannic acid, 3 g/l, was used as an addition agent.

Type of deposit: Burnt, coarse-grained deposit was obtained. "Treeing" was excessive.

Conclusion: None

Experiment No. 17:

Same conditions as were used in experiment No. 15, except corn syrup, 7.5 g/l and gum arabic, 1.1 g/l were used as addition agents.²⁰

²⁰ Creighton, Vol. II, p. 153.

Type of deposit: Very fine-grained deposit.
Although there was very little "treeing" on the edges of the cathode, there was excessive "treeing" on the surfaces between the anodes and cathode.

Conclusion: Anodes were too small and caused the current to be concentrated at the center of the cathode surfaces.

Experiment No. 18:

Time: 3 hr and 50 min

Seal of Quality: 3 oz/ft²

Cathode to anode spacing: 1 3/4 in.

Anode size: 6 1/2 in. x 5 in.

Cathode to anode submerged area (ratio): 1 : 1

Addition agents: 7.5 g/l corn syrup and 1.1 g/l gum arabic.

Current Efficiency: Not measured

Type of deposit: A very fine-grained deposit was obtained and "trees" were absent except for a very small amount at the bottom corners of the cathode.
(Appendix I, Plate I)

Conclusion: The proper use of addition agents is very important in an electrodeposition process.

ELECTROGALVANIZING OF ARMCO IRON WIRE

Experiment No. 19:

Cathode: Armco iron wire $1/8$ in. dia., 3 in. long.

Anode: Circular stainless steel of unknown composition. It was machined from bar stock to the following dimensions:

1. Outside dia.: $1 \frac{3}{4}$ in.
2. Inside dia.: $1 \frac{5}{8}$ in.
3. Length: $2 \frac{1}{2}$ in.

Cathode and anode spacing: $3/4$ in.

Cathode to anode submerged area (ratio): 1 to 10.4

Total area of cathode submerged: 0.982 in.^2

Solution volume: 350 ml

Cell temperature: 25 to 30°C

Addition agent: corn syrup, 7.5 g/l; gum arabic

1.1 g/l

Time: 3 hr 50 min

Seal of Quality: 3 oz/ft^2

Current: 0.14 amps

Voltage: 2.3 v

Current Density: 20 amp/ft^2

Zinc Conc.: 30 g/l

Current Efficiency: Not measured

Type of deposit: A porous but very fine-grained deposit was obtained. (Appendix I, Plates V and VI).

Conclusion: Nickel from the anode contaminated the solution, giving it a green color, and was apparently responsible for the porous zinc coating. As the composition of the bar stock was unknown, it is hard to tell what actually occurred.

Physical Properties of Electrogalvanized Iron:

No. A.S.T.M. tests for adherence and corrosion were made on the galvanized samples. They compared favorably with hot-dipped galvanized iron when they were subjected to bending inasmuch as the hot-dipped sample developed cracks in the zinc coating first. In most cases adherence of the zinc to the base metal was good.

Metallography:

Although there is a zinc-iron alloy a few molecules thick at the zinc coating and base metal interface of an electrogalvanized product, the thick dark line between the zinc and the iron in the photomicrographs of the transverse section of the specimens should not be taken as the zinc-iron alloy. This dark line is a groove which resulted from the etching of the specimens.

CONCLUSIONS AND RECOMMENDATIONS

The electrogalvanizing of iron products using zinc ammonium sulphate leach was a very successful experiment since the desired results were obtained. Although the experiments in this problem were not extensive enough to warrant a comparison with the commercial methods of galvanizing in use today, it is possible that the foregoing method of galvanizing might produce a superior product or at least one as good. This statement is based on several facts. These are as follows:

1. Very fine-grained deposits are obtained by electrolyzing ammoniacal zinc solutions.
2. The thickness of the zinc coating can be easily controlled.
3. Any weight of coating may be applied.
4. The zinc coating is pure and ductile; therefore, it has less tendency to crack when deformed.
5. An excellent bond is obtained between the metal coating and the base metal; therefore, there is less tendency for them to separate.
6. Ammoniacal leaching of zinc calcine eliminates many of the disadvantages of acid leaching.

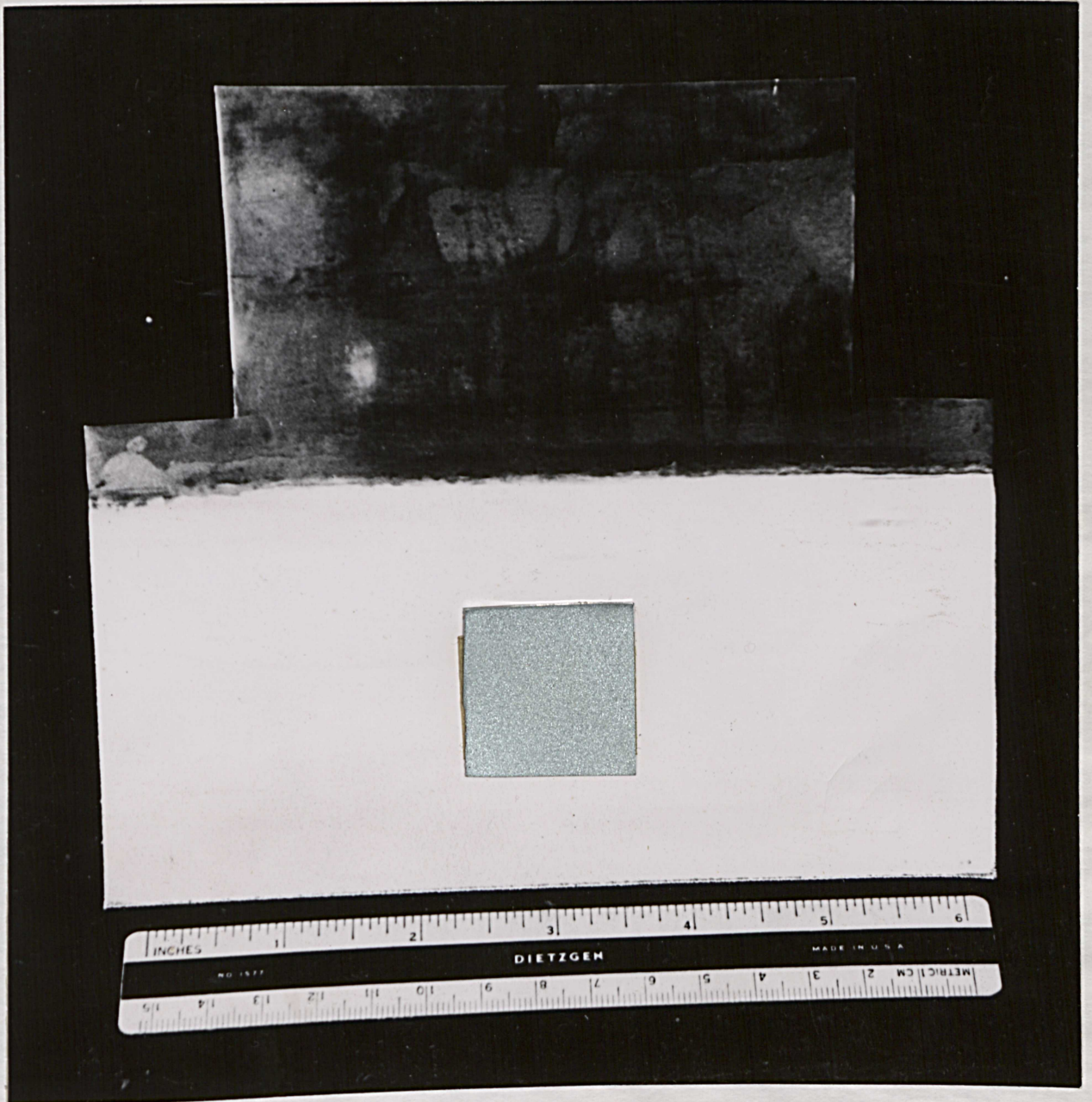
In light of the success met with this problem, the following recommendations are given:

1. The effect of temperature and pH upon the character of the deposit should be determined.
2. Current density, concentration, and temperature should be correlated in order to determine whether or not they affect each other and thereby affect the character of deposit.
3. Other addition agents should be tried.
4. Higher current densities will lessen the time required to obtain a definite weight of deposit. If they are increased in conjunction with the above recommendations, a superior zinc coating should be obtained.

The importance of galvanizing in the prevention of corrosive damage is well known. It is our problem to develop more efficient and more economical methods of combating this costly agent.

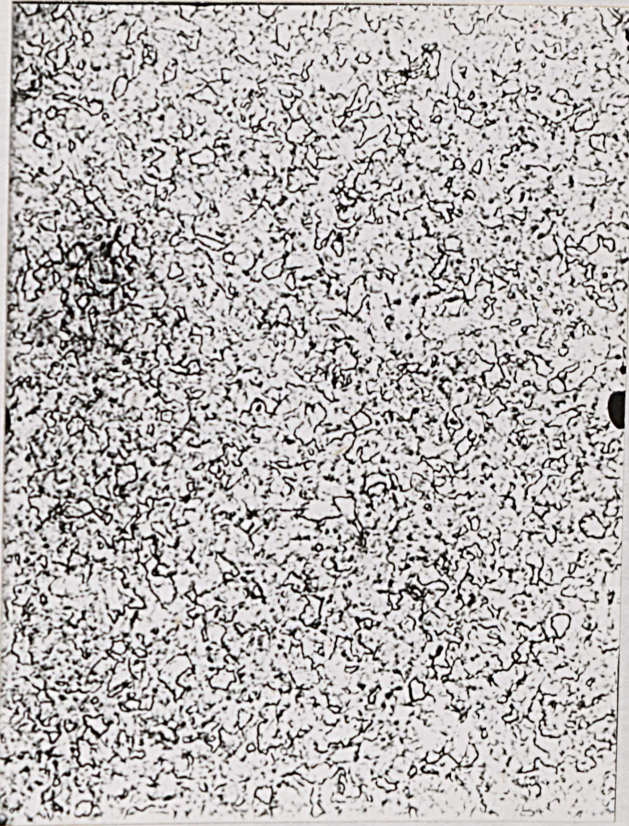
APPENDIX I

Plate I



Galvanized Sheet
Showing Surface of Zinc Coating
Size: $6\frac{1}{2}$ in. x $2\frac{7}{8}$ in.

Plate II



Polished Sample
of Galvanized Surface

Average Grain Size: 576 grains/in.²

Etch: No. 1 Palmerton reagent

250 x

Plate III



Transverse Section
of Galvanized Sheet
Showing Zinc Coating and Base Metal
Etch: No. 1 Palmerton reagent

100 x

Plate IV

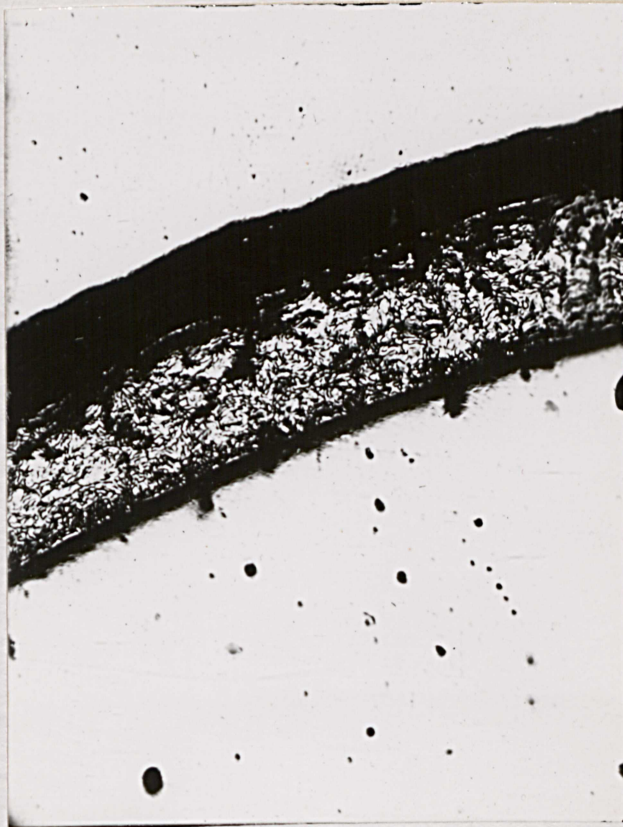


Transverse Section
of Galvanized Sheet
Showing Zinc Coating and Base Metal

Etch: 2% Nital

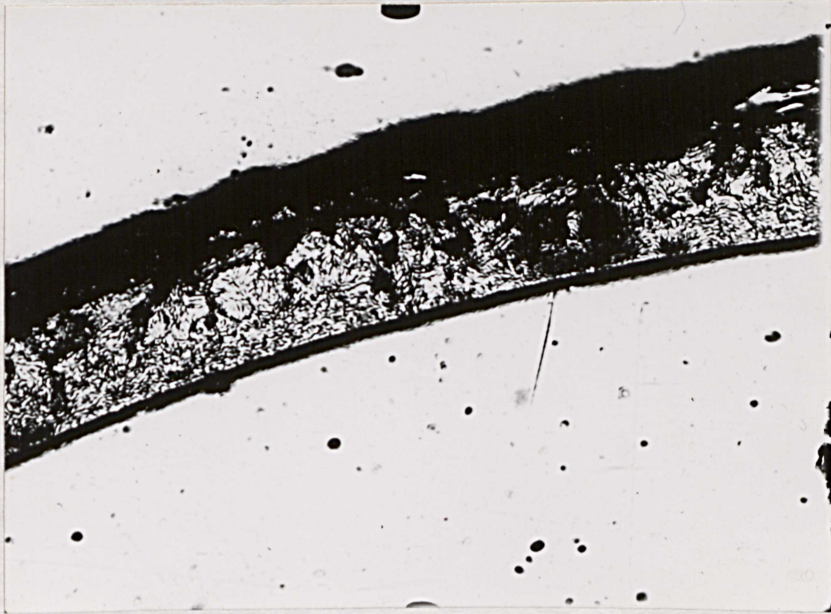
250 x

Plate V



Transverse Section
of Galvanized Armco Iron Wire
After First Reduction
Wire Dia.: 0.125 in.
Galvanized Dia.: 0.132 in.
First Red. Dia.: 0.129 in.
Etch: No. 1 Palmerton reagent
250x

Plate VI



Transverse Section
of Galvanized Armco Iron Wire
After Second Reduction
Second Red. Dia.: 0.1275 in.
Etch: No. 1 Palmerton reagent
250 x

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