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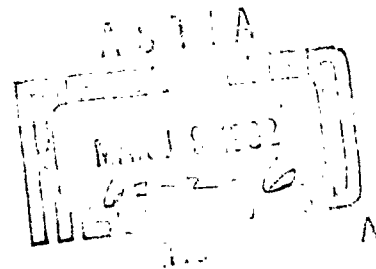


## TECHNICAL REPORT

DEPOSITION OF ALLOYS BY CHEMICAL REDUCTION

By

Jacob Knanishu



Department of the Army Project No. 593-32-006

Ordnance Management Structure Code No. 5010.11.810

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Approved by:

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Laboratory Director

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**ABSTRACT**

A literature survey was made on electroless plating and a historical report on its development is recorded.

The deposition of alloys of nickel, cobalt, and alloy combinations of nickel, chromium and iron were explored, the basis metal being steel, aluminum or magnesium.

Cobalt and nickel were successfully plated on steel and aluminum, as was the plating of nickel on magnesium. Stannate coating of magnesium was also undertaken with success.

## RECOMMENDATIONS

Further investigation should be conducted in the field of electroless plating. Testing of electroless metallic deposits for hardness, corrosion resistance and impact resistance should be explored.

# DEPOSITION OF ALLOYS BY CHEMICAL REDUCTION

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## DEPOSITION OF ALLOYS BY CHEMICAL REDUCTION

### OBJECT

The object of this work is to conduct a literature survey and a laboratory evaluation of various electroless plating processes.

### INTRODUCTION

The study of the deposition of alloys by chemical reduction was initiated so late in the fiscal year that little more than a literature survey was accomplished. Since this project was not funded for the following year, the literature review and preliminary tests are reported for record purposes.

A deposit of nickel by chemical reduction as now known is actually an alloy of nickel and phosphorus. It is believed possible, that by making modifications of the solution composition, a family of alloys such as copper-nickel and nickel-chromium can be produced.

The deposition of cobalt by chemical reduction was developed by A Wurtz<sup>(1)</sup> at the same time as was nickel plating by chemical reduction, but has not gained as much use. It is possible that by altering the nickel bath various alloys could be electrolessly plated. This method of plating can be used to advantage in protective coatings of parts used in the electronic control compartments of rockets and missiles, space instrument packages, and radar equipment, where optimum protection is imperative. Heat transmittance and surface texture of these coatings are important properties which can be incorporated in the above instruments.

Many investigators have reported that by first dipping magnesium in a molten bath of zinc, a coating by electroless deposition could more easily be accomplished. Properly applied, this method produces an adherent coating system on the basis metal. This step (zinc immersion) makes the protective process too complex to be used economically on a production basis, therefore, work has been done to eliminate the immersion phase. However, the greatest problem confronting investigators in plating without a primary dip in zinc is the corrosive attack on the magnesium by the ingredients in the nickel bath.

According to DeLong<sup>(6)</sup>, the solution cannot contain more than traces of sulphates and chlorides. Nickel acid fluoride, sodium hypophosphite, and a pH buffering compound are the main constituents of a successful solution.

As mentioned above, A. Wurtz was the first to reduce nickel compound solutions to nickel by the aid of hypophosphites. The first effort at reduction of nickel produced a precipitate of black powdered nickel. The second trial produced a nickel metal plating on any object that was immersed in the solution, plus a complete coating of the interior of the container that held the solution and object.

It was reasoned by Breteau<sup>(2)</sup> in 1911, during his experiments on nickel and cobalt, that the above reductions were due to the catalytic influence of the metals themselves.

In 1916 F. G. Roux<sup>(3)</sup> was granted a patent on a process similar to that of Breteau.

The potentials of these discoveries were not recognized until 1944, 28 years later, after further work had been done along these lines, when Brenner and Riddell<sup>(4)</sup> of the Bureau of Standards accidentally discovered that exfoliation of a nickel-tungsten alloy was occurring on the interior surfaces of a tube where an insoluble anode was placed. Observing the oxide formation on the anode prompted Brenner and Riddell to add various reducing agents to the solution in an effort to minimize this effect. When, according to Brenner, sodium hypophosphite was used as the reducing agent, the interior of the tube was completely plated. In this process, vigorous gassing occurred with a cathode efficiency of 120-130 percent. This led the researchers to believe that a chemical deposition, as well as electrodeposition, was taking place.

According to Brenner<sup>(5)</sup>, the difference between the process developed by Brenner and Riddell and that of the earlier investigators, Breteau and Roux, was that while the latter was a spontaneous and complete reduction, the former was a catalytic process so controlled that deposition occurred only on the catalytic surfaces. By varying the concentration of the hypophosphite, Brenner found that nickel could be deposited on the surface of iron, nickel, gold, cobalt, palladium and aluminum in this manner.

More recently, with the advent of the use of light metals, considerable work has been done by H. K. DeLong<sup>(6)</sup> on the electroless plating of nickel on magnesium. In his paper, delivered at the 1961 American Electroplaters' Society Convention in Boston, DeLong states that not only does plating on magnesium produce good protection, but also good grounding, conductivity and solderability in the manufacture and use of electronic equipment in the aircraft and missile fields. He also states that tin, nickel, silver, gold and platinum can be applied advantageously as final coats for special uses.

The method outlined by DeLong in his treatise on chemical reduction of nickel onto magnesium appears to be the most successful and simplified method published to date. Previously, many claims had been made in the superlative, but they can hardly be accepted as practical and dependable on a production basis.

For several years, electroless plating of nickel, cobalt and chromium onto steel has been very successful, as noted by Harry J. West<sup>(7)</sup> in a paper entitled, "Electroless Nickel-Barrel Plating". He notes the importance of the pH, the temperature, and the lining used in the plating tank. In a 30 minute operation, an 0.00015 inch thickness deposit can be obtained.

In 1954, Harry J. West, the author of an article entitled "Electroless Nickel Plating On Non-Ferrous Metals", dealt with the chemical reduction method of plating nickel directly onto aluminum, brass, copper and titanium. The solution used was of the alkaline type with the pH held at approximately ten.

Also, in 1954, John D. McLean and Seymour M. Karten<sup>(9)</sup> reported that small fuse parts were successfully nickel plated by chemical reduction for Navy Ordnance. They claimed that, since fuses have recesses and blind holes, the electroless method of plating was the perfect answer.

According to G. Gutzeit and R. W. Landon<sup>(10)</sup>, the main problems in the earlier work of plating by chemical reduction were to increase plating rates, produce a smooth lustrous surface, and a more stable plating solution.

Dr. Abner Brenner<sup>(11)</sup> in a two-part report, conceded that, up to and including the year 1954, only nickel, cobalt, or nickel-cobalt alloys had been deposited by the electroless process in a manner capable of practical application.

In 1955, Harry J. West<sup>(12)</sup> reported that the corrosion resistance as determined by the salt spray (fog) test of a thin film of chromium on steel deposited by the electroless process, compared equally with the same coating thickness (0.0002 inches) of chromium on steel.

#### PROCEDURE AND RESULTS

Since the time allotted for work on the electroless plating project was short, attention was focused on magnesium, because techniques of plating on magnesium are the least known and are presently important.

With the proper formulation and operation, acid and alkaline baths lend themselves well to the chemical reduction process. In plating directly onto magnesium, only the alkaline bath may be utilized, since acid causes extreme etching and thus prevents an adherent coating from being formed.

Various combinations of composition materials were tried, with varying results. The increase or decrease in the temperature of the solutions resulted in variations of quality and quantity of coatings produced. Experiments with baths similar in composition to that proposed by DeLong<sup>(13)</sup> proved most successful.

The specimen to be plated was first chemically etched in a chromic-acid-nitric-acid solution, shown in the table below, until the surface was completely renewed, the amount of etching being proportional to the time of immersion.

Chromic Acid (CrO <sub>3</sub> )	60 gms/liter
Nitric Acid (70% HNO <sub>3</sub> )	90 ml/liter

Immersion in the solution was followed by a water rinse, then a hydrofluoric acid immersion for 10 minutes as prescribed by DeLong. For high aluminum-magnesium alloy content, 220 ml/liter of 70% hydrofluoric acid was used. For alloys under 5%, a weaker solution (54 ml/liter of 70% hydrofluoric acid) was used. After the specimen was thoroughly rinsed in water, it was transferred to the plating solution. Good to excellent results were obtained by using the above etching procedure and the electroless plating bath as follows:

	<u>Per Liter</u>
Basic Nickel Carbonate (2NiCO <sub>3</sub> · 3Ni(OH) <sub>2</sub> · 4H <sub>2</sub> O) -	10 gms
Hydrofluoric Acid (as 70% HF)	6 ml
Citric Acid (C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> )	5.5 gms
Ammonium Acid Fluoride	15 gms
Sodium Hypophosphite (NaH <sub>2</sub> PO <sub>2</sub> · H <sub>2</sub> O)	20 gms
Ammonium Hydroxide (30% NH <sub>4</sub> OH)	30 ml
pH Operational Range (colorimetric)	4.5-6.8
Operating Temperature	170-180 F
Agitation	mild
Rate of Deposition (inches)	about 0.0008/hr

In the preparation of 1 liter of the solution, the basic nickel carbonate was slowly added to the hydrofluoric acid in approximately 100 ml of water to form the nickel acid fluoride. This step requires considerable agitation. After the reaction was complete, the remainder of the chemicals were added in the order they appear in the above table.

Water was added to effect dissolution. Additional water was then added to produce 1 liter of solution. The solution was adjusted to pH 6.5 and then filtered. Operating the bath at 170° - 180° F, the rate of deposition was found to be 0.0006 inches per hour.

A bath similar to that employed by Brenner and Riddell was prepared, and the plating of nickel onto steel and aluminum was performed with excellent results. Cobalt was successfully deposited on steel panels by chemical reduction, using the solution formulation and procedure outlined by Brenner and Riddell(4).

An electroless chromium plating solution was then made. This solution was similar in composition to that used by Harry J. West(12). In the operation procedure of electroless chromium plating, catalytic initiation is necessary to promote the plating process. This initiation was brought about by placing a catalyst metal, such as iron or aluminum, in the solution and allowing a momentary contact with the part to be coated. Once this process has begun, the initial deposit produced catalytic action, and plating continued. This method produced a satisfactory deposit. The formulation of the solution was as follows:

Chromium Fluoride	64 gms/gal of solution
Sodium Chloride	4 gms/gal of solution
Sodium Citrate	32 gms/gal of solution
Sodium Hypophosphite	32 gms/gal of solution
Water - to make 1 gallon of solution	
Temperature	160° - 190° F

A stannate bath for the deposition of tin directly onto magnesium was made, as follows:

Potassium Stannate	5%
Potassium Pyrophosphate	5%
Potassium Hydroxide	.75 - 1.5%
Sodium or Potassium Acetate	1%

The bath was operated for 20 minutes at 195° - 200° F.

The procedure followed was:

The test panels were degreased, then placed in a chromic acid cleaning solution, rinsed in water, and put into an alkaline cleaning solution until a bright surface was obtained, the alkaline cleaning composition being:

Sodium Hydroxide	2-4 oz/gal
Sodium Carbonate	3-4 oz/gal
Wetting Agent	0.1 oz/gal
Operating Temperature	200°F ± 10°F
Immersion Time	10-15 minutes

The panels were removed from the alkaline cleaning solution after a 10-15 immersion, and rinsed thoroughly. They were then placed in the stannate solution for 20 minutes. Excellent coatings were obtained. However, some of the panels showed a pearlescent color due to too thin a coating. It was noted that, in order to obtain a good coating, replenishing of the solution with tin salts, and maintaining the optimum temperature range of 195° - 200°F was necessary. Mechanical agitation also favored good deposition.

Stannate coated panels were placed in the salt spray (20%) cabinet in order to compare the protection afforded the magnesium base with that of the Dow 17 treatment<sup>(14)</sup>. Some of the stannate coated panels were given various organic protective top coats, and they, too, were subjected to the salt spray test. After 888 hours, the Dow 17 treated panels with an organic top coat showed no signs of a breakdown. The stannate pretreated panels had failed at 508 hours, and although the exposure time may be considered good, the comparative value of stannate pretreatment is much less than the Dow 17 treatment in anti-corrosion qualities.

The following bath<sup>(4)</sup> was used successfully to plate nickel on to both steel and aluminum:

Nickel Chloride	30 gms/liter
Sodium Hypophosphite	10 gms/liter
Ammonium Chloride	50 gms/liter
Sodium Citrate	100 gms/liter
Ammonium Hydroxide to a pH of 8-10	
Temperature	190°F

Electroless plating of cobalt was carried out in a bath<sup>(14)</sup> of the following composition and operating technique:

Cobalt Chloride	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	30 gms/liter
Sodium Hypophosphite	$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	20 gms/liter
Sodium Citrate	$\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 5\text{-}1/2 \text{H}_2\text{O}$	35 gms/liter
Ammonium Chloride	$\text{NH}_4\text{Cl}$	50 gms/liter
Ammonium Hydroxide	$\text{NH}_4\text{OH}$ for neutralizing bath	
pH		9-10
Temperature		190°-200°F

Catalytic initiation was necessary to start deposition. Less noble metals such as iron or aluminum can be used.

The nickel plating bath as formulated by DeLong is not only tolerant to magnesium, but also to other metals such as aluminum, iron, stainless steel, copper, tin and brass. Its ability to plate on the above metals makes it a prospective plating solution when combination couplings of these metals are already in an assembly prior to plating.

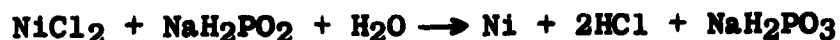
The quality of the coatings can be improved<sup>(15)</sup> by using a heat treatment at 750 C or higher (1382 F). The high heat makes the coating compact and adherent and causes the diffusion of the iron and nickel to produce a nickel-iron alloy.

## DISCUSSION

It is important that the constant decomposition of the surface of the basis metal be avoided. Therefore, in order to obtain a complete and continuous coating, the solution must be so formulated that it will deter the oxidation of the surface and still promote chemical reduction of the metal salts used in the plating bath. When magnesium is the basis metal in a chemical reduction process, the nickel must be in the form of nickel-acid-fluoride. Chloride or sulphate ions must be absent from the bath because they produce corrosion action on magnesium. Using a conventional formulation for electroless nickel plating on magnesium, a poorly adherent and irregular coating was produced. This was due to the corrosive action of the chloride ions during deposition. In the areas where the nickel plating was somewhat continuous, the appearance was dull.

It has been shown<sup>(4)</sup> that hypophosphites are excellent reducing agents in the electroless plating process. Investigators have used this compound in many chemical reduction processes of various metals such as nickel, cobalt, tungsten and chromium. Its reducing action takes place in the presence

of catalytic metals. In the electroless plating of nickel, the following reactions occur:



In the presence of water, the hypophosphite undergoes oxidation and the nickel is reduced. Since these two ingredients are constantly changing chemically, they must be added as replenishing materials in order that the reaction can continue. In an alkaline solution, the rate of nickel deposition is only slightly affected by the concentration of nickel ions. This rate of deposition is mainly a function of hypophosphite concentration. The reaction which determines the deposition rate in alkaline solution is the reaction of hypophosphite to produce hydrogen, which, in turn, reduces the nickel on the catalytic surface<sup>(4)</sup>.

When the basis metal does not possess catalytic properties, the reaction must be initiated by momentarily injecting into the solution a catalytic metal which contacts the basis metal. Once this is done, plating begins and the deposited metal takes over the role of a catalyst and plating continues as long as the solution is replenished with the salts of the plating metal, and with a sufficient amount of hypophosphite to reduce these salts.

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