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METHOD OF ELECTROLESS NICKEL PLATING AND PLATING BATHS THEREFOR

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ABSTRACT OF THE DISCLOSURE

Electroless nickel plating is conducted using an aqueous acid plating bath in which a single compound, nickel fluoborate, $(NiBF_4)_2$, is the source both of the nickel ions and of the buffer for control of the pH of the bath and sodium hypophosphite is the reducing agent for the nickel ions.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates to electroless nickel plating of metal surfaces, more particularly to an improved method and plating solutions therefor.

Electroless nickel plating was discovered by Brenner and Riddell and reported by them in J. Res. Nat. Bur. Standards, 39, 385 (1947). They used aqueous acid plating baths formed of a solution in water of a soluble nickel salt, such as nickel chloride and nickel sulfate, as the source of the nickel ions, sodium hypophosphite as the reducing agent for the nickel ions, and a buffer compound such as sodium acetate, sodium hydroxyacetate and sodium citrate as the control for the pH of the plating bath. Practice in the electroless nickel plating art has followed Brenner and Riddell in the use of separate compounds for providing the nickel ions and the buffer in the plating baths.

It is an object of the present invention to provide an improved electroless nickel plating method in which a single compound is used to provide both the nickel ions and the buffer in the plating bath.

It is also an object to provide an electroless nickel plating method which is effective to directly plate metals which are anti-catalysts or poisons for the plating method as it has been heretofore practiced.

It is a further object to provide new plating solutions for electroless nickel plating which are simpler and more economical than heretofore known.

The above and other objects are accomplished following the present invention by conducting electroless nickel plating with aqueous acid plating baths in which the single compound, nickelous fluoborate, $Ni(BF_4)_2$, is both the source of the nickel ions and of the buffer for control of the pH of the plating bath, and sodium hypophosphite is the reducing agent for the nickel ions.

The new plating solutions are prepared by simply adding sodium hypophosphite to a water solution of the nickelous fluoborate and stirring to dissolve the hypophosphite. The concentration of the nickelous fluoborate in the plating solutions may vary considerably depending on the magnitude of the surface area to be plated and the thickness of the plating desired. In general proportions of nickelous fluoborate in the range of from about 5 to 50% by weight of the solution may be used, with a preferred range of proportions being from about 10 to 20% by weight. The sodium hypophosphite is used in the plating solutions in an amount sufficient to reduce the nickel ions during the plating. In general proportions of sodium hypophosphite in the range of from about 1 to 10% by

weight of the solution may be used, with a preferred range of proportions being from about 2 to 4% by weight.

Electroless plating with the new plating solutions is generally to be conducted at elevated temperatures, for which a preferred temperature range is from about 150° F. to the boiling point of the solution or about 220° F. and an optimum range is from about 180° to 200° F. The pH of the plating solutions during the metal deposition may range from about 2 to 6.

The electroless nickel plating system of the invention is characterized by the tendency to increase in rate of metal deposition as the pH of the bath drops into the lower end of the pH scale such that at a pH of 2, metal deposition occurs at a practical rate. Conversely, in the Brenner and Riddell type plating system, the rate of metal deposition tends to decrease as the pH of the bath drops into the lower end of the pH scale and at a pH of 2, metal deposition no longer occurs at a practical rate. The plating method of the invention is also distinguished over the Brenner and Riddell type plating system in a higher rate of metal deposition.

Additional advantages of the new electroless plating method over the Brenner and Riddell type plating method are simplicity and economy in the plating bath, only the reducing agent, the nickel fluoborate and water being required for replenishing of the bath; easier control of the system both in rate characteristics and the amount or thickness of metal deposited, due to fewer constituents in the system, and greater stability, in that the system has a higher tolerance to contaminants which normally cause premature reduction of the nickel ions and less susceptibility to erratic fluctuations of operating conditions, such as "hot spots" during the plating reaction.

A further advantage of the new electroless plating system is that it possesses the ability to deposit the metal directly on metals, such as lead, tin and cadmium, etc., which normally are anti-catalysts or poison the plating reaction in the Brenner and Riddell type plating system.

Various metals may be plated with nickel by the method of the invention, for example, iron, steel, stainless 18–8 steel, zinc, copper, brass, bronzes, aluminum, tin, lead and cadmium. Plating of the metal surface is by chemical reduction through reaction of the nickel salt with the hypophosphite, with nickel ions being reduced in the plating solution. The rate of deposition of nickel on the metal surface in the plating increases with increase in the bath temperature. For plating baths which are solutions in water of 20% by weight of nickelous fluoborate and 4% by weight of sodium hypophosphite and are at a temperature in the range of from about 180° to 215° F., the rate of metal deposition onto the immersed metal surface is in the range of about 1 to 3 mils per hour.

In the practice of the method of the invention, the metal surface to be plated should be in a clean, degreased condition, free from scale, as is understood and practiced in the plating art. The metal article to be plated is simply immersed in the plating bath for the time required to attain the desired thickness of plating on the metal surface. Support of the article for plating may be accomplished in any suitable manner, as by holding with the hand or with a mechanical means.

The invention is further illustrated by the following specific examples of the practice thereof.

EXAMPLE 1

A length of high carbon steel key stock, ¼ x ¼" square cross-section, was cleaned for plating by washing with a detergent, washed with water, dipped in aqueous hydrochloric acid and again washed with water. The length of cleaned steel surface, for the major portion thereof, was immersed in an aqueous plating bath formed

of a solution in water of 12.3% by weight of nickelous fluoborate and 2.5% by weight of sodium hypophosphite and maintained at a temperature of approximately 180° F. Immersion time for the steel surface in the plating bath was approximately 6½ hours. Micrometer measurement of the metal deposited on the steel surface was 6.25 mils.

EXAMPLE 2

A tin surface and a cadmium surface were plated with nickel by immersion in plating baths formed of a solution in water of nickelous fluoborate and sodium hypophosphite in proportions similar to those in Example 1. The temperature of the plating baths during the immersion of the metal surfaces was approximately 180° F.

It has been also found that the method of the invention is effective to deposit a nickel coating on non-metal surfaces such as glass, polytetrafluoroethylene (fused) and polymethylmethacrylate.

While the invention has been described herein with reference to certain specific embodiments thereof, it is to be understood that such are to be taken by way of illustration and not in limitation except as may be defined in the appended claims.

What is claimed and desired to be secured by Letters Patent of the United States is:

1. A method of plating nickel on a metal surface by chemical reduction which comprises immersing the metal surface in a plaing bath consisting essentially of a solution in water of nickelous fluoborate and sodium hypophosphite and maintained at a temperature in the range of from 150° F. to about the boiling point thereof, said plating bath containing from about 5 to 50% by weight of nickelous fluoborate and from about 1 to 10% by weight of sodium hypophosphite, and holding the metal surface immersed in said plating bath for a time sufficient to deposit nickel thereon.

2. A method as defined in claim **1**, wherein the plating bath is maintained at a temperature in the range of from about 180° F. to 200° F.

3. A method as defined in claim **1**, wherein the plating bath contains from about 10 to 20% by weight of nickelous fluoborate and from about 2 to 4% by weight of sodium hypophosphite.

4. A solution for use as the plating bath in chemical reduction nickel plating and consisting essentially of a solution in water of from about 5 to 50% by weight of nickelous fluoborate and from about 1 to 10% by weight of sodium hypophosphite.

5. A solution as defined in claim **4**, in which the concentration of nickelous fluoborate is from about 10 to 20% by weight and of the sodium hypophosphite is from about 2 to 4% by weight.

6. A method as defined in caim **1**, wherein the metal is lead.

7. A method as defined in claim **1**, wherein the metal is tin.

8. A method as defined in claim **1**, wherein the metal is cadmium.

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