United States Patent

Zuntini et al.

[15] 3,637,474

[45] Jan. 25, 1972

[54] **ELECTRODEPOSITION OF PALLADIUM**

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[22] Filed: **Aug. 26, 1968**

[21] Appl. No.: **755,386**

[30] **Foreign Application Priority Data**

Sept. 8, 1967 Switzerland 12612/67

[52] **U.S. Cl.** **204/47**

[51] **Int. Cl.** **C23b 5/24,** C23b 5/46

[58] **Field of Search** ....2?4/47, 43, 44, 109, 110; 106/1; 117/130 E

[56] **References Cited**

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[57] **ABSTRACT**

An electroplating bath for the deposition of palladium from a palladium-urea complex. A method for depositing palladium from a chemical solution containing a palladium-urea complex by passing an electric current through the solution, whereupon palladium deposits on the surface of a workpiece or a cathode.

9 Claims, No Drawings

ELECTRODEPOSITION OF PALLADIUM

This invention relates to an electroplating solution and process for electrodepositing palladium. More particularly, this invention relates to the electrodeposition of palladium onto the surface of another metal which serves as the workpiece or cathode.

Palladium has been electrodeposited from a number of aqueous solutions. The most popular palladium-electroplating solution utilizes palladium in the form of a salt, particularly palladium diamino-dinitrite, known as palladium "P" salt as the source of palladium ions. However, deposits obtained from the known plating baths heretofore used suffer from the deficiency of being porous in the thin 2.5 to 5 micron coatings range which is the most desirable range from a commercial standpoint. The porosity is due primarily to the fact that the deposits from known plating solutions are highly stressed. Porosity results in corrosion of the underlying metal.

Another difficulty with known electroplating solutions for depositing palladium is that they produce a dull deposit requiring further treatment to brighten.

It has now been discovered that a stable pore-free deposit in the thicknesses customarily used for contacts, connectors, printed circuit boards, etc., can be obtained by the electrodeposition of palladium from a solution containing palladium in the form of a palladium-urea complex. Surprisingly enough, the deposit is not only pore-free in the thin coatings generally used, but remains so when used for heavy, thicker deposits. Additionally, the deposits of palladium are bright, and indeed can actually be made "white" if a further ingredient, namely a sulfite salt is used in combination with the palladium-urea complex.

A palladium-urea complex salt can be obtained by reacting palladium chloride, or sodium palladium chloride or other soluble palladous salts with a slight excess of urea. A salt containing approximately 36 percent palladium metal precipitates, which is soluble in alkaline solutions containing urea.

The palladium-urea complex dissolved in a solution containing urea can be used directly for electrodepositing palladium. The solution must be alkaline and that is easily accomplished by using ammonium hydroxide or similar alkalies. However, it is preferable to add other salts to the solution to improve the conductivity of the solution. Such salts can be any water-soluble salt which does not cause a precipitation in the bath by reacting with the palladium ions or with urea. For example, phosphates are suitable. Furthermore, it is desirable to add complexing or chelating agents to the solution, such as the sodium salt of ethylenediamine tetra-acetic acid to prevent the contamination of the bath with ions of the metal on which the deposit is effected. In most cases the palladium is deposited over copper, copper alloys or nickel and nickel alloys and there is a certain amount of drag-in of contaminating ions. Those ions are chelated by EDTA and similar compounds thus preventing codeposition of ions of the metal on which palladium is being deposited.

The pH of the plating bath is not critical, but should be maintained alkaline, preferable above 8 and preferably by the addition of ammonium hydroxide, although other alkalies are suitable.

The current density is not critical and can be varied. One skilled in the art will recognize that the current density can be varied greatly depending upon the amount of agitation and density in the solution. However, the preferred current density range is about 0.1 to about 1.2 amp. dm.^{2}.

The temperature is not critical and in general it is preferred to operate a temperature of from about 40° to about 60° C.

The following examples will demonstrate how the plating bath and method works. Their purpose is not, however, to limit the invention because one skilled in the art could propose many variations in bath composition using this novel palladium salt.

EXAMPLE 1

A bath was made up as follows:

Palladium (as the urea complex)	10 g./l.
Sodium nitrite	7 g./l.
Ammonium phosphate	80 g./l.
Urea	8 g./l.
Ammonium salt of EDTA	10 g./l.
Ammonium hydroxide to pH of	9.5

At a temperature of 50° C. and a current density of 0.3 amp. dm.^{2} bright palladium deposits were obtained.

EXAMPLE 2

To the solution described in example 1, without the sodium nitrite, was added 25 g. of sodium sulfite (anhydrous).

At a current density of 0.5 amp./dm.^{2}, bright and pore-free deposits were obtained at thickness of 2 to 10 microns. The deposits were whiter than those obtained in example 1.

The palladium content of this bath was increased to 15 g./l. by adding additional palladium-urea complex. Using high agitation, satisfactory deposits were obtained at 1.2 amp./dm.^{2}.

EXAMPLE 3

A bath made up as follows:

Palladium (as urea complex)	2 g./l.
Ammonium phosphate	50 g./l.
Urea	5 g./l.
Sodium sulfite	5 g./l.
Ammonium hydroxide to pH	8.5

This bath gave bright, white deposits at a current density of 0.1 amp./dm.^{2} and a temperature of 55° C.

The quantity of sodium sulfite was increased to 75 g./l. and the urea palladium compound was added to the limit of its solubility. The soluble sulfite compound can be present in a quantity such that the sulfite ion concentration is between approximately 1.5 and approximately 70 grams per liter of solution. Under these conditions, bright deposits were obtained at current densities well over 1.0 amp. dm.^{2}.

We claim:

1. An electroplating bath for depositing palladium therefrom comprising an aqueous alkaline solution of a urea complex of palladium wherein the palladium content of the bath derived from said complex is at least about 2 grams per liter.

2. The bath as claimed in claim 1 wherein the pH of the solution is 8.0 or greater.

3. The bath as claimed in claim 1 wherein a soluble sulfite compound is added in a quantity such that the sulfite concentration is between approximately 1.5 and approximately 70 grams per liter of solution.

4. The bath as claimed in claim 1 wherein a salt of ethylene diamine tetra-acetic acid is present in an amount sufficient to complex contaminating metal ions.

5. A method of electrodepositing palladium on a workpiece which comprises passing an electric current through a chemical solution wherein the workpiece serves as a cathode, said chemical solution comprising an aqueous alkaline solution of a palladium-urea complex wherein the palladium content of the bath derived from said complex is at least about 2 grams per liter.

6. A method as claimed in claim 5 wherein the chemical solution contains a soluble sulfite compound in a quantity such that the sulfite concentration is between approximately 1.5 and approximately 70 grams per liter of solution.

7. A method as claimed in claim 6 wherein a salt of ethylene diamine tetra-acetic acid is present in an amount sufficient to complex contaminating metal ions.

8. A method as claimed in claim 5 wherein the electric current passing through the solution is such that a current density of about 0.1 to about 1.2 amp. dm.^{2} is maintained.

9. A method as claimed in claim 5 wherein the temperature of the solution is maintained from about 40° to 60° C.