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(54) **A method of high speed electroplating palladium and palladium electroplating solution therefor.**

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**EP-A-0 020 020**
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**US-A-3 920 526**
**US-A-3 972 787**
**US-A-4 093 520**
**US-A-4 098 656**
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## Description

The invention relates to a method of high speed electroplating palladium and palladium electroplating solution therefor.

The dramatic increase in the price of gold over the past several years has given rise to new methods and equipment in the electroplating field and attempts to use substitute metals such as palladium through selective plating. Specialized plating equipment such as described, for example, in U.S. Patent Nos. 3,723,283 and 3,819,502, and others of a similar nature, utilize chemical or mechanical masks which limit the metal deposits to the specific required area while leaving other areas free of the metal. The use of equipment of this type makes possible a reduction in the amount of metal deposited by a factor of 10 or above compared with equipment and methods utilizing overall plating techniques and results in a like reduction in the cost of the electroplated item produced.

The use of such plating process requires very high speed plating and high speed requires high current densities preferably above 100 ASF (~1,000 A/m$^2$). A discussion of some of the problems and advantages of high speed plating is found in U.S. Patent 3,879,269.

Attempts have been made to plate palladium from high speed plating equipment with various baths, however, the deposits are either burned and matte gray or they are bright to semi-bright and highly stressed and exhibit surface microcracks which are visible only under the microscope at high power. These cracks can be visible in the deposit right out of the plating bath or they become visible later or after the deposit has been permitted to stand at room temperature for about a day or so. Much has been written about these cracks and it is generally attributed to the codeposition of hydrogen with palladium, and after the hydrogen is emitted from the deposit cracks will appear. The industry desires to have palladium deposits that are crack-free at usable current densities in high speed plating. The current densities required for high speed plating in today's technology range from about 100 to 2,000 ASF (~1,000—20,000 A/m$^2$) and higher. High speed plating equipment employs the jet plating principle where the solution is sprayed out onto the surface being plated with a jet stream, to provide very vigorous agitation. It is also possible to supply vigorous agitation without the jet stream, but by moving the solution very rapidly past the part being plated through the use of a pump and/or by moving the parts rapidly through the solution.

Prior art palladium plating solutions are exemplified by U.S. Patents 3,972,787 and 4,092,225, U.S. published patent application B450,499 and Russian Patent 354,010.

U.S. Patent 3,972,787 discloses a number of palladium plating baths which contain quaternized pyridinium compounds as brighteners. The first four specific baths are disclosed as having a current density range from 0.1 to 40 ASF (~400 A/m$^2$) except for Example 5 which was 0.1 to 50 ASF (~1—500 A/m$^2$). All of these baths are unsuitable for high speed plating. The patent also discloses that at 20 g/l palladium it is possible to plate at 500 to 1,000 ASF (~5,000—10,000 A/m$^2$) at 50°C, but without the brighteners the panel is dull gray above 4 ASF (~40 A/m$^2$).

The Russian patent discloses palladium chloride, ammonia ions and ammonia phosphate at a pH of 6.1 and 6.7. The usable current density range of these solutions is 6 to 8 ASF (~60—80 A/m$^2$), and they cannot be used in high speed plating.

The published U.S. application uses palladium ammonium chloride and free ammonia at a pH of at least 8.8. These baths contain no phosphate or carbonate anions.

Patent 4,092,225 adds palladium as $Pd(NH_3)_2(NO_2)_2$ and tetrapotassium pyrophosphate to form a bath at a pH of 8.5 to 11 and discloses that the solutions are suitable for rack plating at 2—50 ASF (~20—500 A/m$^2$).

US—PS 3,920,526 describes non-porous ductile palladium which is obtained by electrodeposition from a high chloride bath comprising about 16 to about 32 g/l of $Pd(NH_3)_2Cl_2$ (palladosammine chloride), about 65 to 250 g/l of $NH_4Cl$ and sufficient aqueous $NH_3$ to provide a pH of at least 8.8. The chloride bath according to the above cited US—PS does not contain any phosphates or carbonates.

DE—A—30 45 968 discloses an aqueous electroplating bath for the preparation of ductile, non-porous palladium deposits which contains 5 to 50 g/l, preferably 30 to 40 g/l, palladiumamino chloride and/or palladiumamino bromide, 10 to 150 g/l sulfamic acid and 10 to 150 g/l ammonium chloride and has a pH of about 6.5 to 10.0, preferably about 8.0 to 9.0. Also this bath does not contain any phosphate or carbonate. The electroplating can be carried out at a current density of 10 to 1076 A/m$^2$.

This invention involves the discovery that palladium plating solutions containing phosphates or carbonates can be formulated without quaternized pyridinium brightening agents and operated under certain plating conditions to produce acceptable crack-free lustrous palladium deposits within specific high current density ranges suitable for high speed plating. The invention is predicated upon fairly well defined ranges of solution ingredient concentrations as well as operating conditions. The object is to obtain an acceptable lustrous deposit which is crack free over a practical high current density range suitable for use in high speed plating, such as high speed jet plating equipment and cathode agitation systems.

The features of the palladium electroplating solution and the methods of electroplating according to the present invention are defined in claims 1, 4 and 5. Preferred features are defined in the dependent claims.

When plating palladium from any known bath

that applicants are aware of, the current density can be raised stepwise until a point is reached above which typical burning of the deposit will take place. A burnt palladium deposit has a powdery dark gray appearance that is almost black and totally matte. Our invention consists of the discovery of a specific set of operating parameters and solution parameters at high current densities to produce lustrous crack free palladium deposits where one would otherwise expect the deposit to be burnt or the deposit cracked.

In high speed jet stream plating, agitation is generally quite violent. Velocities of 200 to 250 inches (~508—635 cm) per second are not uncommon. Due to these high velocities, by way of a jet stream, the actual velocities at the cathode can vary considerably and therefore it is necessary to have a fairly wide current density range for practical commercial purposes.

The velocities reported herein and in Figure 5 are obtained by measuring flow to the jets in cubic inches per second and dividing this figure by the total jet area measured in square inches. The cathode surface being plated is also larger than the jet stream impinging upon it; therefore, the velocity figures do not represent velocity impingement over the entire cathode surface nor what actually takes place on the cathode surface. The extent of agitation at the cathode depends generally on the geometry of the system, such as the size of the cathode, the particular mask being employed, and the distance between the jet outlets and the cathodes etc., as will be apparent to those skilled in the art. Thus, there will generally be large variations in agitation over the surface of the cathode, and this is the reason why wide current density is desired. The experiments reflected in the drawings were made using a fairly large specific area (about 1/4" square ≈1,6 $cm^2$) which is one of the more difficult cathode areas to plate requiring a fairly wide current density window or delta because of large variations of agitation at the cathode. With smaller cathode areas, a more narrow window or delta can be tolerated, such as a delta of about 200 ASF (~2,000 $A/m^2$). Delta as used herein means the range in which acceptable deposits can be obtained. For example, in Figure 1 at 45 g/l of ammonium phosphate the acceptable range is from 750 ASF to 2000 ASF (~7,500—20,000 $A/m^2$) and the delta or difference is 1250 ASF (~12,500 $A/m^2$), and at 75 g/l the delta is 750 ASF (~7,500 $A/m^2$).

A delta of at least 750 ASF (~7,500 $A/m^2$) is generally desirable and a delta of 500 ASF (~5,000 $A/m^2$) would generally be acceptable depending on the plating speeds and type of cathode being plated. These deltas lend diversification to the solutions to permit plating of large cathode areas as well as small areas which might only require an ASF delta of about 200 (~2,000 $A/m^2$) without changing solutions. Many objects to be plated also have variable current density requirements, again requiring a fairly wide current density window. The current density range selected will also depend on the current density requirements of the article to be plated. Some articles may require a range from 750 to 2000 ASF (~7,500—20,000 $A/m^2$) while others 500 to 1500 ASF (~5,000—15,000 $A/m^2$), while other articles can even be plated, such as small round dots, with a delta of 200 ASF (~2,000 $A/m^2$). With cathode rod agitation equipment utilizing cathode movement, it is also advantageous to have as wide a window as possible. At an agitation rate of about 3 inches (7.62 cm) per second at 100 ASF (~1,000 $A/m^2$) this window, for lustrous crack free deposits, should advantageously have a delta of at least 50 ASF (~500 $A/m^2$).

U.S. patent No. 3,972,787 discloses the use of certain brighteners in baths containing palladium chloride and solutions 4 and 5 include phosphates. The patent also states that without the brighteners, the panels are dull gray above 4 ASF (~40 $A/m^2$). Contrary to this disclosure, the applicants have found that when solution 4 is plated at a current density between 250 and 500 ASF (~2,500—5,000 $A/m^2$) and solution 5 at a current density between 200—300 ASF (~2,000—3,000 $A/m^2$) without the brighteners, the deposits are crack free and the luster is fair. Below the lower current densities, the deposits are dull gray and above the higher current densities they are burnt. These solutions have current density deltas of 250 and 100 (~2,500 and 1,000 $A/m^2$) and are not too practical for use in plating with high speed jet plating equipment. Plating from solutions 4 and 5 without the brighteners with cathode movement equipment gave no useful current density range and bright deposit could not be obtained. A current density delta of at least about 50 (~500 $A/m^2$) when operating at a current density of 100 ASF (~1,000 $A/m^2$) is almost essential for practical high speed cathode movement palladium plating. The patent also discloses that when the palladium metal content of the solution of the examples is increased to 20 g/l using $2\times10^{-4}$ mols/liter of the brightener, bright deposits can be obtained from 500 to 1000 ASF (~5,000—10,000 $A/m^2$). Such deposits, however, will exhibit surface microcracks and be unacceptable.

The applicants have discovered that by adjusting the palladium metal content to about 10 g/l and above, maintaining the phosphate content above 7.5 g/l and below 150 g/l (calculated as ammonium phosphate), and maintaining the pH to between about 7 and 9, that these solutions can be successfully employed in high speed plating equipment at current densities above 100 ASF (~1,000 $A/m^2$) to produce lustrous crack free deposits so long as adequate agitation or the flow rate is maintained.

Figures 1 through 6 graphically depict certain of the operating windows with the variations in the phosphate content, the palladium content, the temperature, flow rate (jet stream agitation) and cathode rod agitation.

As shown in Figure 1, an operating window exists above 7.5 g/l of ammonium phosphate up to 150 g/l. At zero ammonium phosphate there was no operating window and the deposits were

dull over the entire current density range. The operating window broadens and a very acceptable broad operating window is found at 45 g/l of ammonium phosphate, and the most acceptable range for the phosphate content, under the standard conditions set for the experiment depicted in Figure 1, is from about 80 g/l of ammonium phosphate to about 20 g/l. At 75 g/l the deposits were somewhat brighter. At 150 g/l the current density range was about 650 to 800 ASF (~6,500—8,000 A/m$^2$) and, although the deposits were brighter, cracking took place in the deposit.

Although ammonium phosphate was used in these experiments, all phosphate compounds tried to date were found to be similarly operable so long as they were soluble in the bath. Potassium pyrophosphate can also be used, for example. The presence of sufficient phosphate or carbonate anion is the important factor and the concentration of other phosphates will have to be adjusted to give equivalent anion concentration with respect to the experiment using ammonium phosphate. Any solution soluble carbonate or bicarbonate compound such as sodium or potassium carbonate can be used to supply the carbonate ion.

Although there is no drawing depicting variations in the operating windows based on the pH of the plating solution, the operating pH range is between about 7 and below 9. When utilizing a solution containing 30 g/l of palladium, 45 g/l of ammonium phosphate, a temperature of 60°C and a flow rate of approximately 208 inches (528 cm) per second, there was no operating range or window at all at pH of 6.5. At a pH of 9 the operating window narrowed to about 50 ASF (~500 A/m$^2$) and ammonia driven off. Using these same conditions but lowering the temperature of the bath to 40°C an operating window of 500—1500 ASF (~5,000—15,000 A/m$^2$) is obtained. This shows that by variation of any one of the important parameters a usable current density range for high speed plating can be obtained or the range can be moved in one direction or another.

Figure 2 shows that variations in the operating window by altering the palladium metal content. At pH of 7.5 and a flow rate of about 250 inches (635 cm) per second and a palladium content of 7.5 g/l, there was no operating window at all at temperatures of 40°C, 50°C and 60°C. It is apparent from the graph that amounts of palladium metal in excess of 45 g/l can be used, but such large amounts would be unnecessary.

Figures 3 and 4 show that there is little effect on the width of the current density window with temperature change under the set conditions of the experiments. The graphs do show that temperature changes can be advantageously employed to shift the lower and upper limits of the window. Temperature does have a profound effect however upon pH change. With the compositions shown in Figure 3 at a pH of 9, the solution was not useable for high speed plating, however, the same composition at a temperature of 40°C gave an operating window of 500—1500 ASF (~5,000—15,000 A/m$^2$). Below 500 ASF (~5,000 A/m$^2$) the deposits were unuseable and above 1500 ASF (~15,000 A/m$^2$) the deposits were burnt.

Figure 5 shows the current density variations due to flow rate with jet stream high speed equipment while Figure 6 shows the current density windows using cathode rod agitation under the conditions noted.

The data contained in Figures 1, 2, 3 and 5 were obtained with high speed jet stream plating equipment under the conditions shown and with cathode agitation equipment in Figures 4 and 6. Experiments such as those shown in Figures 1, 2 and 3 will show substantially the same results with cathode agitation but not the same numbers. The windows or current density delta will be smaller, but this is acceptable as noted above with cathode agitation and other high speed plating process.

It must be borne in mind that the graphs are only illustrative of the invention being based on one specific set of conditions with only one variable. For example, the operating window of Figure 1 will not be the same if the set conditions are changed to a palladium content of 20 or 45 g/l. In such a case, it may be advantageous to change the pH, the flow rate or the phosphate or carbonate content to obtain the optimum operating window, and this can readily be accomplished by routine experimentation.

The palladium metal can be supplied to the solution by any compound capable of forming a palladium tetraamino complexion. Examples of such compounds include: $Pd(NH_3)_4^{++}X$ where X is an anion such as chloride, bromide, acetate, sulfate and nitrate. The addition of palladium metal as palladium tetraamino chloride is preferred.

**Claims**

1. A palladium electroplating solution comprising an ammoniacal solution of a palladium tetraamino complex ion with a palladium metal concentration above 10 g/l, a pH between 7 and 9, said solution being free of quaternized pyridinium brightening agents and comprising a phosphate in a concentration of above 7.5 g/l and below 150 g/l calculated as ammonium phosphate.

2. The solution of claim 1 wherein the phosphate anion is supplied by ammonium phosphate or potassium pyrophosphate.

3. The palladium electroplating solution of claim 1, wherein the phosphate concentration is between 20 and 80 g/l.

4. A method for high speed electroplating of palladium to produce lustrous crack-free palladium deposits, said method comprising plating the palladium on a substrate with high speed plating equipment at 50 ASF (~500 A/m$^2$) and above by using the electroplating solution of one of claims 1, 2 or 3.

5. A method for high speed electroplating of

palladium to produce lustrous crack-free palladium deposits, said method comprising plating the palladium on a substrate with high speed plating equipment at 100 ASF (~1,000 A/m²) and above by using an electroplating solution comprising an ammoniacal solution of a palladium tetraamino complex ion with a palladium metal concentration above 10 g/l, a pH between 7 and 9, and carbonate ion in a sufficient concentration to permit plating of palladium at 100 ASF (~1,000 A/m²) and above, said solution being free of quaternized pyridinium brightening agents.

6. The method of claim 5, characterized in that the carbonate ion is supplied by any solution soluble carbonate or bicarbonate compound.

7. The method of one of claims 4, 5 or 6, characterized in that the solution or substrate is agitated.

8. The method of claim 7, characterized in that the palladium deposits are plated at a current density delta of at least 200 ASF (~2,000 A/m²) and jet agitation speeds above 125 inches (317.5 cm) per second.

9. The method of claim 8, characterized in that the current density delta for jet stream agitation is 500 ASF (~5,000 A/m²).

10. The method of claim 7, characterized in that the palladium deposits are plated at a current density delta of at least 50 ASF (~500 A/m²) at a cathode agitation above 3 inches (7.6 cm) per second.

**Patentansprüche**

1. Palladium-Elektroplattierlösung, welche eine ammoniakalische Lösung eines Palladium-tetraamino-Komplexions mit einer Palladiummetall-Konzentration von über 10 g/l umfasst, einen pH zwischen 7 und 9 aufweist, wobei die genannte Lösung frei von quaternisierten Pyridinium-Glanzbildnern ist und ein Phosphat in einer Konzentration von oberhalb 7,5 g/l und unterhalb 150 g/l, berechnet als Ammoniumphosphat, umfasst.

2. Lösung nach Anspruch 1, worin das Phosphatanion von Ammoniumphosphat oder Kaliumpyrophosphat herrührt.

3. Palladium-Elektroplattierlösung nach Anspruch 1, worin die Phosphatkonzentration zwischen 20 und 80 g/l liegt.

4. Verfahren zum Schnellplattieren von Palladium zur Bildung glänzender, rissfreier Palladiumüberzüge, wobei dieses Verfahren eine Palladiumplattierung auf einem Sustrat mit Hilfe einer Schnellplattierungsausrüstung bei 50 ASF (ca. 500 A/m²) und darüber unter Verwendung einer Elektroplattierlösung gemäss Ansprüchen 1, 2 oder 3 umfasst.

5. Verfahren zum Schnellplattieren von Palladium zur Bildung glänzender, rissfreier Palladiumüberzüge, wobei dieses Verfahren eine Palladiumplattierung auf einem Sustrat mit Hilfe einer Schnellplattierungsausrüstung bei 100 ASF (ca. 1.000 A/m²) und darüber unter Verwendung einer Elektroplattierlösung, welche eine ammoniakalische Lösung eines Palladium-tetraamino-Komplexions mit einer Palladiummetall-Konzentration von über 10 g/l, einem pH zwischen 7 und 9, und Carbonation in ausreichender Konzentration, um eine Palladiumplattierung bei 100 ASF (ca. 1.000 A/m²) und darüber zu erhalten, umfasst, wobei die Lösung frei von quaternisierten Pyridinium-Glanzbildnern ist.

6. Verfahren nach Anspruch 5, dadurch gekennzeichnet, dass das Carbonation von einer in der Lösung löslichen Carbonat- oder Bicarbonatverbindung herrührt.

7. Verfahren nach einem der Ansprüche 4, 5 oder 6, dadurch gekennzeichnet, dass die Lösung oder das Sustrat bewegt wird.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, dass die Palladiumüberzüge bei einem Stromdichte-delta von mindestens 200 ASF (ca. 2.000 A/m²) und Strahlbewegungsgeschwindigkeiten von über 125 inches (317.5 cm) pro Sekunde aufgebracht werden.

9. Verfahren nach Anspruch 8, dadurch gekennzeichnet, dass das Stromdichte-delta bei Strahlbewegung 500 ASF (ca. 5.000 A/m²) beträgt.

10. Verfahren nach Anspruch 7, dadurch gekennzeichnet, dass die Palladiumüberzüge bei einem Stromdichte-delta von mindestens 50 ASF (ca. 500 A/cm²) bei einer Kathodenbewegung von über 3 inches (7,6 cm) pro Sekunde aufgebracht werden.

**Revendications**

1. Solution pour dépôt électrolytique de palladium comprenant une solution ammoniacale d'un ion de complexe palladotétrammine avec une concentration en palladium métal supérieure à 10 g/l, un pH compris entre 7 et 9, cette solution étant exempte d'agent de brillantage au pyridinium quaternisé et comprenant un phosphate à une concentration supérieure à 7,5 g/l et inférieure à 150 g/l calculée sous forme de phosphate d'ammonium.

2. Solution selon la revendication 1 dans laquelle l'anion phosphate est fourni par du phosphate d'ammonium ou du pyrophosphate de potassium.

3. Solution d'électrodéposition de palladium selon la revendication 1 dans laquelle la concentration en phosphate est comprise entre 20 et 80 g/l.

4. Procédé de dépôt électrolytique à vitesse élevée de palladium pour la production de dépôts de palladium brillants exempts de fissures, ce procédé comprenant le dépôt électrolytique du palladium sur un substrat avec un équipement d'électrodéposition à vitesse élevée à 50 ASF (env. 500 A/m²) et au-dessus, par l'emploi de la solution d'électrodéposition de l'une des revendications 1, 2 ou 3.

5. Procédé de dépôt électrolytique à vitesse élevée de palladium pour produire des dépôts de palladium brillants et exempts de fissures, ce procédé comprenant le dépôt électrolytique du palladium sur un substrat avec un équipement d'électrodéposition à vitesse élevée à 100 ASF

(env. 1000 A/m$^2$) et au-dessus, par l'emploi d'une solution d'électrodéposition comprenant une solution ammoniacale d'un ion de complexe palladotétrammine avec une concentration en palladium métal supérieure à 10 g/l, un pH entre 7 et 9 et un ion carbonate à une concentration suffisante pour permettre le dépôt électrolytique du palladium à 100 ASF (env. 1000 A/m$^2$) et plus, ladite solution étant exempte d'agent de brillantage au pyridinium quaternisé.

6. Procédé selon la revendication 5, caractérisé en ce que l'ion carbonate est fourni par une solution quelconque soluble d'un composé de carbonate ou de bicarbonate.

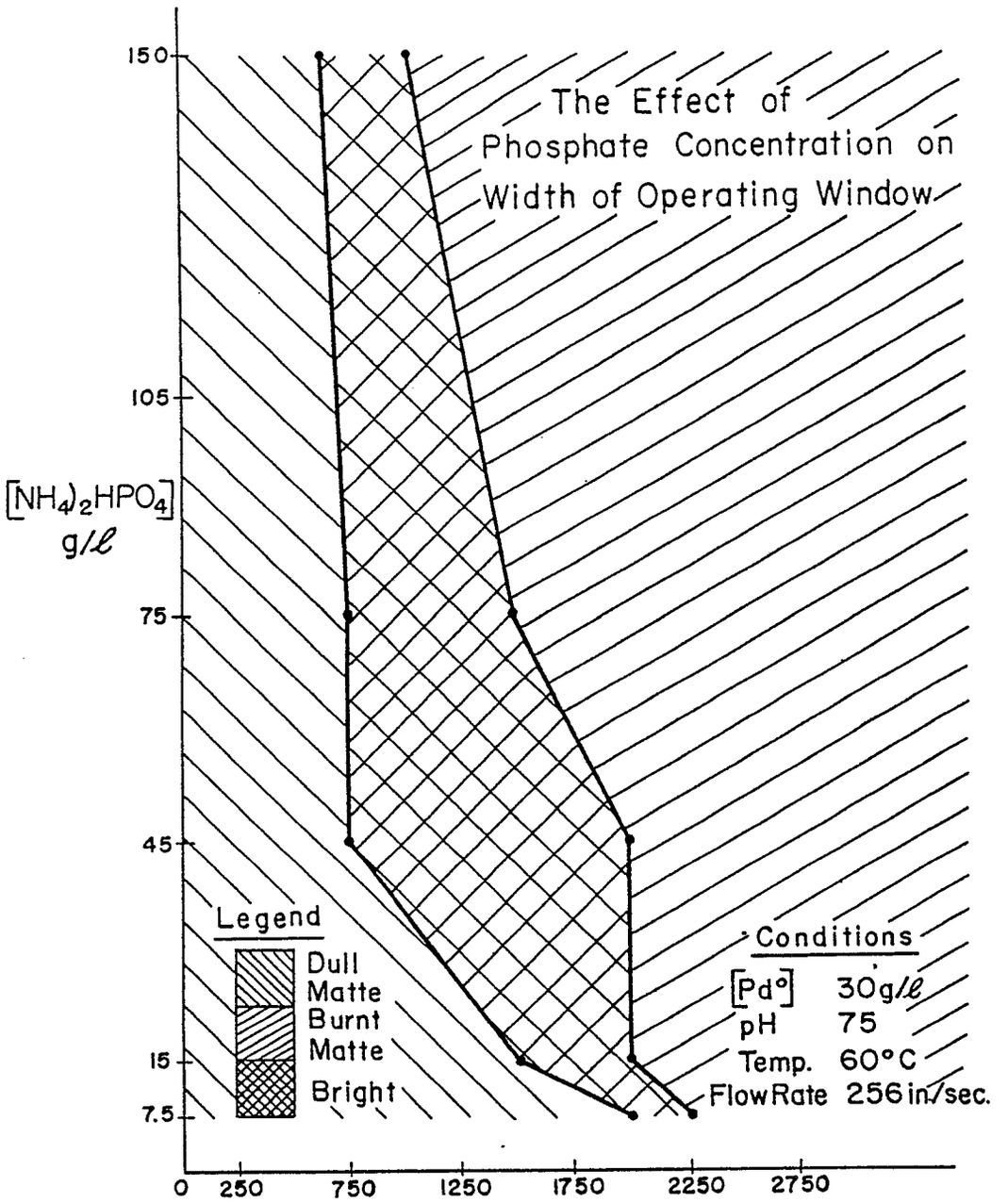
7. Procédé selon l'une des revendications 4, 5 ou 6 caractérisé en ce que la solution ou le substrat sont agités.

8. Procédé selon la revendication 7, caractérisé en ce que les dépôts de palladium sont déposés par électrolyse avec une variation de la densité de courant d'au moins 200 ASF (env. 2000 A/m$^2$) et des vitesses d'agitation par jet supérieures à 125 pouces (317,5 cm) par seconde.

9. Procédé selon la revendication 8, caractérisé en ce que la variation de la densité de courant pour une agitation par jet projeté est de 500 ASF (env. 5000 A/m$^2$).

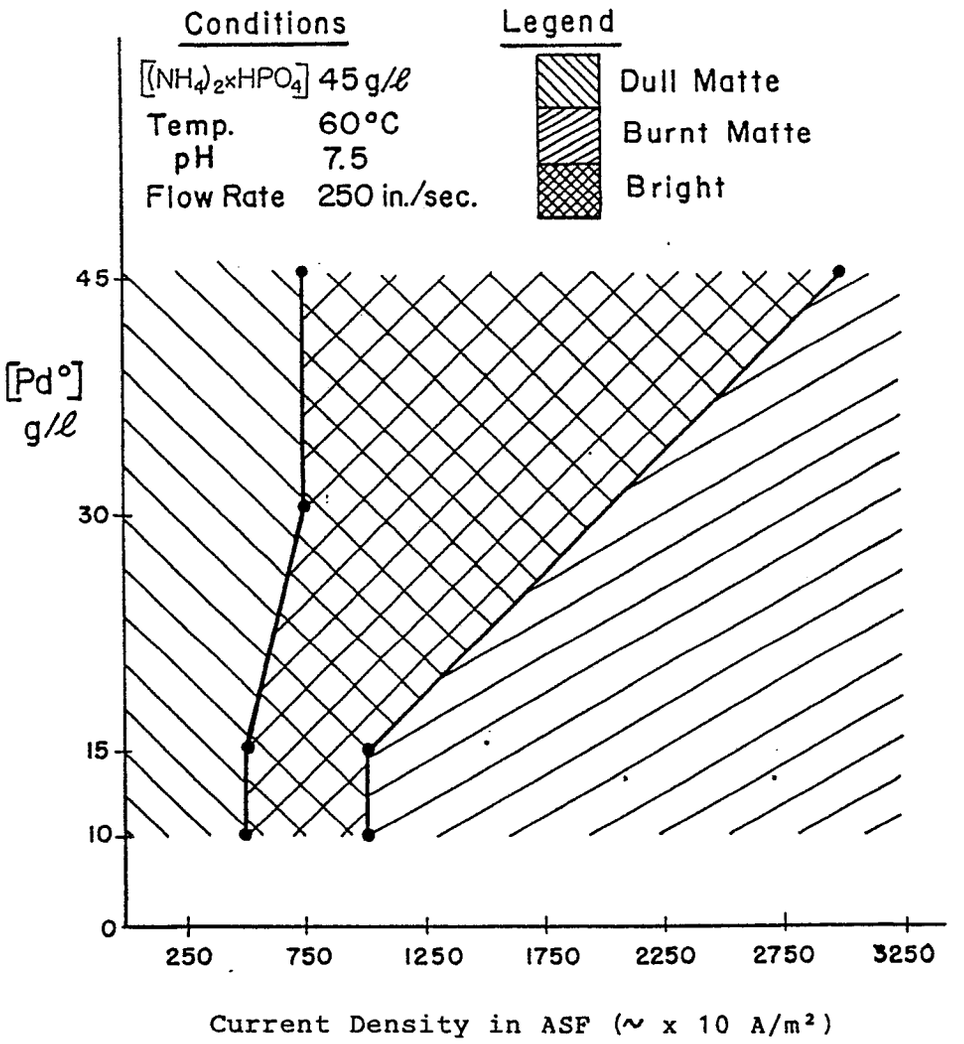
10. Procédé selon la revendication 7 caractérisé en ce que les dépôts de palladium sont déposés par électrolyse avec une variation de la densité de courant d'au moins 50 ASF (env. 500 A/m$^2$) avec une agitation de la cathode supérieure à 3 pouces (7,6 cm) par seconde.

FIG. 1


The Effect of
Phosphate Concentration on
Width of Operating Window
150
105
75
45
15
7.5
[NH4)2HPO4]
g/ℓ
Legend
Dull
Matte
Burnt
Matte
Bright
Conditions
[Pd°] 30g/ℓ
pH 75
Temp. 60°C
FlowRate 256in/sec.
0
250
750
1250
1750
2250
2750
Current Density in ASF (~ x 10 A/m²)

# FIG. 2

The Effect of Palladium Concentration on Width of Operating Window



# FIG. 3

The Effect of Temperature on
Width of Operating Window

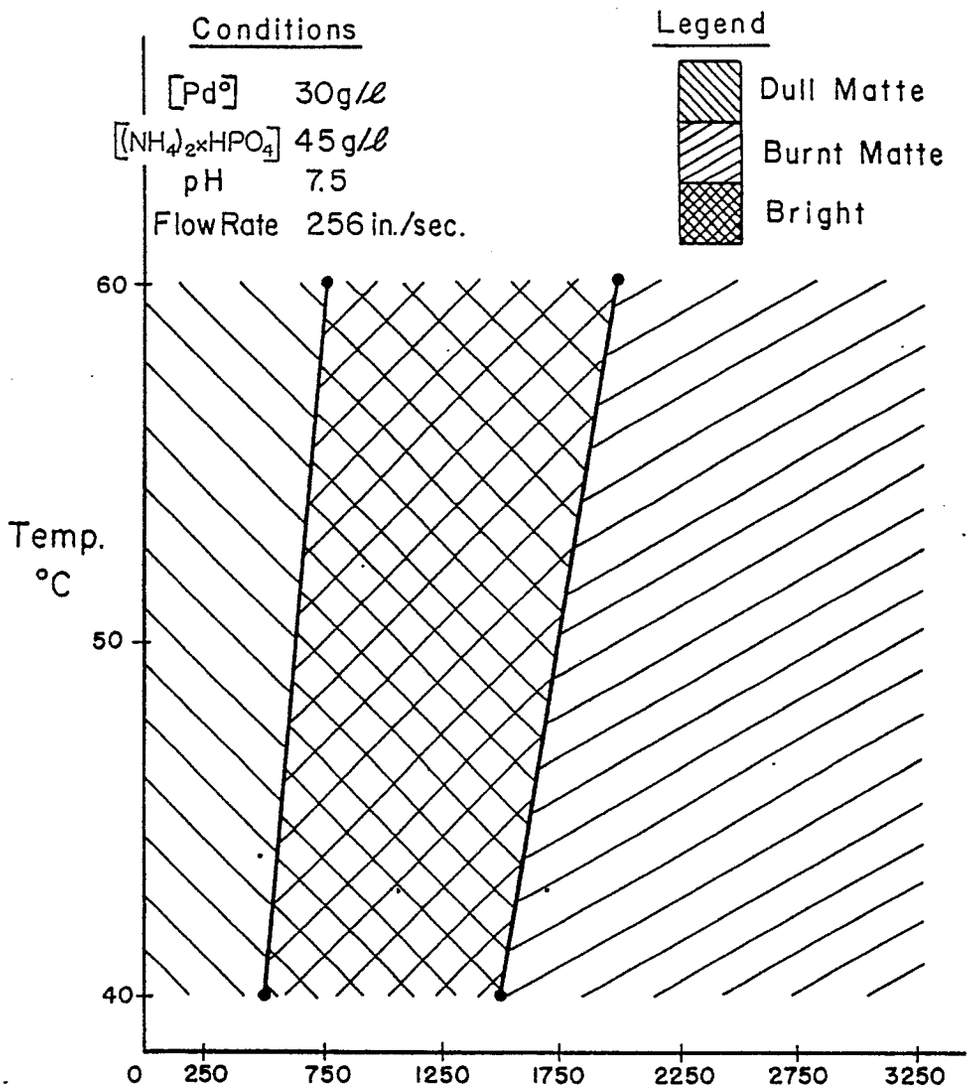


FIG. 4

The Effect of Temperature on Width of Operating Window

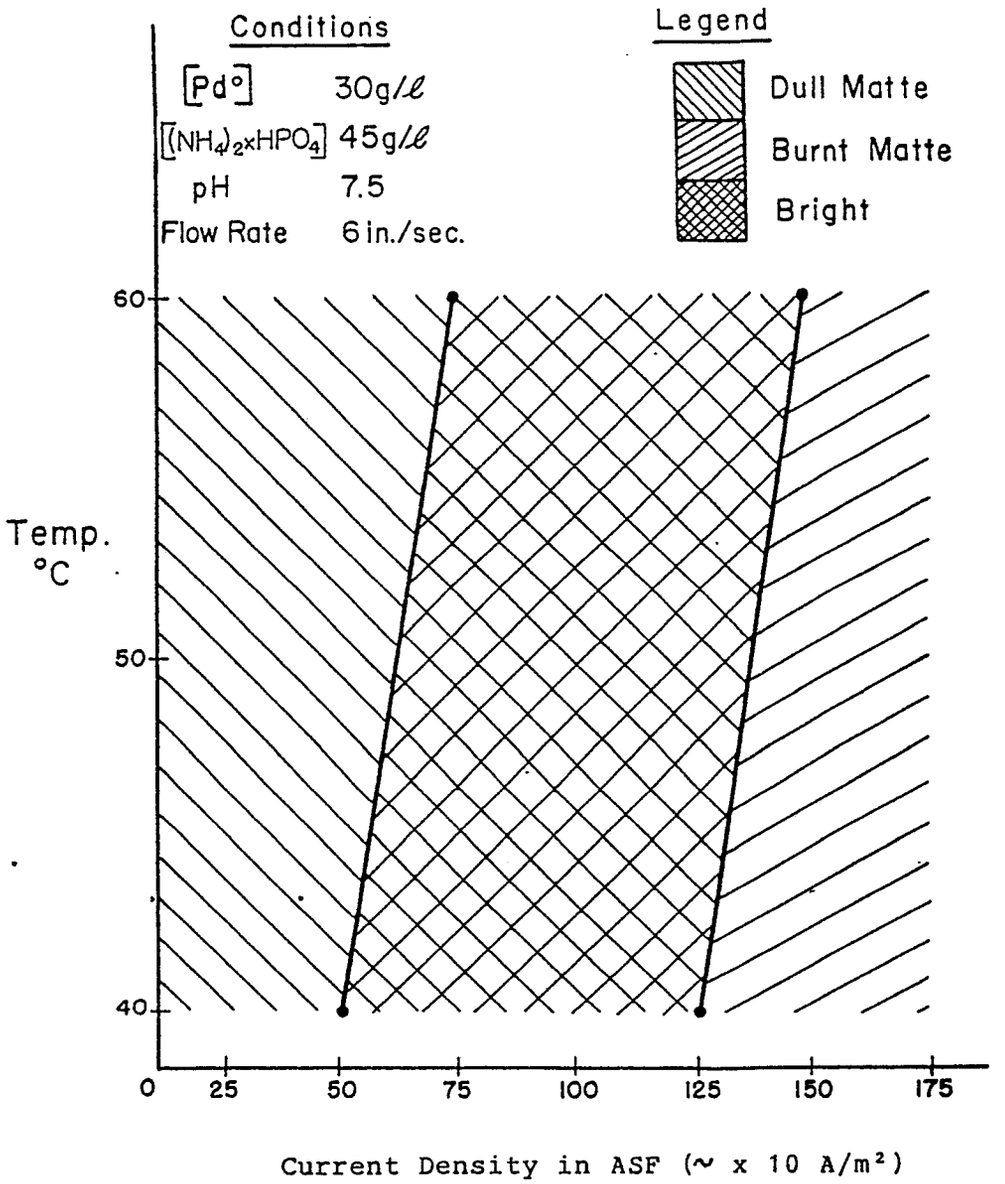
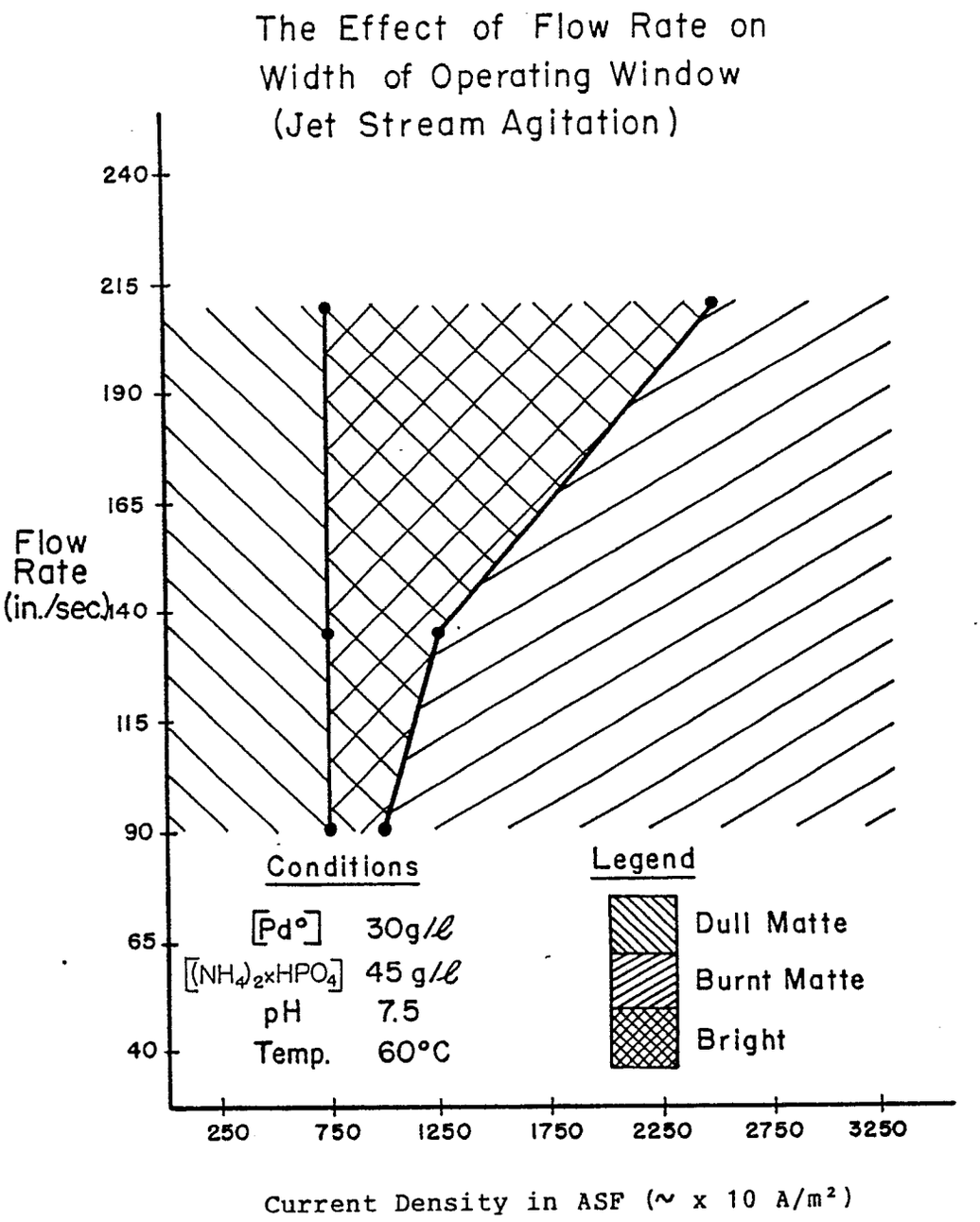


FIG. 5


The Effect of Flow Rate on
Width of Operating Window
(Jet Stream Agitation)
Flow Rate (in./sec.)
240
215
190
165
140
115
90
65
40
Conditions
[Pd°] 30g/ℓ
[(NH4)2xHPO4] 45 g/ℓ
pH 7.5
Temp. 60°C
Legend
Dull Matte
Burnt Matte
Bright
250
750
1250
1750
2250
2750
3250
Current Density in ASF (~ x 10 A/m²)

# FIG. 6

The Effect of Agitation on
Width of Operating Window