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특집 : Electroless Plating of Copper

Recent Progress in Electroless Plating of Copper

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Abstract: In this article, the recent developments in electroless plating of copper, electroless bath formulation and effect of plating parameters have been reviewed. Cyanide free electroless baths are now being developed and studied due to the various environmental concerns. Various organic chemicals such as complexing agents, reducing agents, and additives such as poly-alcohols and aromatic ring compounds have been added to copper plating baths for promising results. The effects of various reducing and complexing agents, bath conditions like additives, bath pH, and composition have been summarized. Finally the applications of the electroless plating of copper and latest developments have been overviewed for further guidance in this field.

Keywords: Electroless, additives, plating, copper, complexing agents, temperature

1. Introduction

Electroless plating is known for hundreds of years. It is a process of deposition of thin layer of metals, salts, oxides and other compounds used in various industrial and technological applications. The deposition of precious metals like gold and silver were already used in early civilizations. Wurtz in 1844 discovered the first nickel electroless coating using hypophosphite as a reducing agent. Later in 1946, Brenner and Riddell developed the process and proposed the operating conditions for the nickel electroless plating. They included various reducing agents including sodium hypophosphite to obtain a controlled nickel plating. William Blum coined the term ‘Electroless’ for this autocatalytic process because of no need of supplying external current into the electrolyte bath.[1)] In electroless technique, a noble metal is deposited from its salt on a catalytic active surface of a less noble metal. On the contrary the electroplating involves supply of an external current for reaction to happen. Apart from composition and organic additives, electroplating involves a number of parameters like current, pulse type, frequency and duty cycle of pulse, etc., making the process more complex.[2-7)] While in electroless, the reaction is catalyzed by employing a suitable reducing agent which supplies the electron for reduction reaction and metal is deposited over the substrate.[1,8)] The reactions can be shown as below:

$$\text{Metal ion } (M^+) + \text{Reducing agent (Red)} \rightarrow \text{Metal Deposited} + \text{Oxidized product (Ox)} \quad (1)$$

There are two reactions in electroless plating:

$$\text{Metal deposition: } M^+ + e \rightarrow M \quad (1a)$$

$$\text{Oxidation: Red} + H_2O \rightarrow \text{Ox} + H^+ + e \quad (1b)$$

Here, M: Metal, e: electron, Red: Reducing agent, Ox: Oxidized product

The process is electrolytic as well as electroless in nature as shown in Fig. 1.

The thickness of electroless coatings can reach up to 10 to 200 μm. Electroless plating is more useful than electroplating, for example, the possibility of producing coatings with uniform thickness, depositing material even in deep recess/vias, ability to produce very thin layers, excellent step coverage, independent of the sizes, shape or conductivity of the substrate and absence of need for electrical contacting of wafers during deposition.[9,10)] Significant advancement occurred in electroless plating of Cu, Ni, Au, Ag, Pd, Sn, etc., for industrial applications, however, the various operating parameters and bath conditions are not fully understood.[10)]

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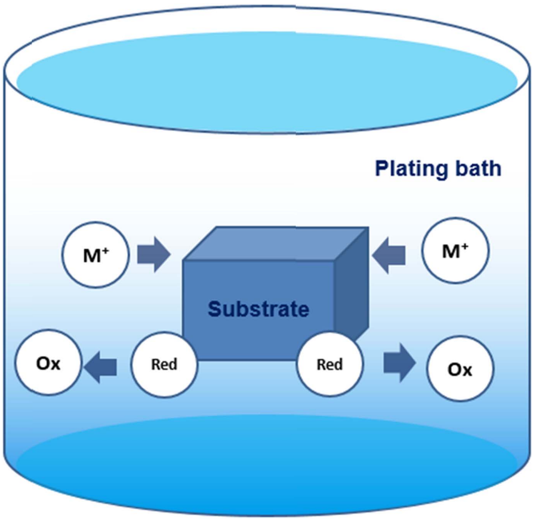

Fig. 1. Schematic representation of electroless metal deposition.

2. Electroless Copper Plating

Copper is one of the oldest element known in the history. In Latin, copper is termed as c*uprum* the Iceland of Cyprus dates back to 11000 years.[11] In the late 18th century, copper was picked up by many scientists like Ampere, Faraday and Ohm for their discoveries in electricity and magnetism. Pure copper is soft, malleable and ductile with very high thermal and electrical conductivity.[10,11] Electroless copper coatings provide protection for common metal surface exposed to corrosion and wear. Electroless copper is the material of today. As a coating, it is used in several industrial applications ranging from aerospace, automotive, electronics, telecommunications, information technology, food processing, beauty care products, nuclear engineering, petrochemicals, plastics, power transmission, printing, pump valves, textiles etc..[12] Electroless copper is being extensively used in microelectronics and packaging of devices, for interconnects in ultra large scale integration (ULSI), IC fabrication and EMI shielding because it doesn't require vacuums or high temperature for thin metal film deposition due to their high conductivity, resistance to electro migration and no requirement for vacuum and high temperature.[10-12]

3. Plating Baths

3.1. Traditional baths

Traditional electroless plating baths are based on reducing agents based on formaldehyde and its derivatives. This type of baths have two major drawbacks, such as, (1) formaldehyde baths operates around alkaline pH values (> 11).[10,13] (2) Aldehydes are volatile, flammable and pose health hazard to human beings. Aldehydes have significant environmental impact and continuous exposure to formaldehyde causes severe skin rashes, eye infections, difficulty in coughing and breathing.[10-14]

To overcome these drawbacks, people have used many electroless copper baths based on non-formaldehyde chemicals, e.g., hypophosphite, sodium bisulfate ($NaHSO_3$), glyoxylic acid, the sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$), borane-dimethylamine complex, Co(II), Fe(II),[15-23] etc. Sodium hypophosphite is most attractive due to its excellent bath stability, easy control, and lower cost. However, the hypophosphite may oxidize on prolong storage and may lead to the reduction in deposit quality. For, alkaline baths the oxidation of hypophosphite can be minimized by the addition of catalytic ions like Ni^{2+} or Pd^{2+} ions.[19,21-24] This increases the efficiency of the process but limits the conductivity of the deposits due to the impurities of Ni and Pd incorporated.[19]

3.2. Methyl sulphonic acid (MSA) baths

MSA baths are getting more attention in the formulation of electrolytic baths in electroless as well as electroplating deposition of metals. MSA is the reducing acid which minimizes the oxidation of the metal ions. Procell et al. discovered that alkyl sulphonic acids form highly soluble metal salts in water producing clear solutions.[24] MSA based baths become popular during 1980's for different metal plating baths and is an excellent choice for various plating applications.[1,8-11,25,26] MSA acid is a transparent liquid having chemical formula CH_3SO_3H characterized by

(i) Transparent slight yellowish
(ii) Soluble in water and sparingly soluble in benzene
(iii) Insoluble in paraffins
(iv) High conductivity better than HCl and H_2SO_4
(v) Excellent stability
(vi) Less toxic and safe to handle
(vii) Biodegradable, easy disposal

The deposits produced from MSA baths are of high quality, high adhesion strength, smooth free from dendrites and porosity. The superiority of MSA over other plating baths arises due to the fact that it can be operated at room temperature, excellent bath life and conductivity, and biodegradability.[25,26]

3.3. Complexing Agents

The complexing agents in electroless copper baths are very important for good quality deposits. Complexing agents minimize the formation of copper to copper hydroxides ($Cu(OH)_2$) in alkaline pH range.[17] Complexing agents stabilized the baths and increases bath life. Addition of complexing agents in a small quantity increases in plating rate. The mixed potential theory of electroless plating states that the overall reaction of the electroless copper process is given by two following half-reactions:

Cathodic reaction:

$$Cu[L]x +2 +2e^{-} \rightarrow Cu + xL \quad (2)$$

Anodic reaction:

$$2HCHO + 4OH^{-} \rightarrow 2HCOO^{-} + H_2 + 2H_2O + 2e^{-} \quad (3)$$

During the electroless process, the two reactions occur simultaneously on a catalytic surface. The most common complexing agents for electroless copper are Ethylenediaminetetraacetic acid (EDTA), trisodium citrate, malic acid, lactic acid, triethanolamine, sodium potassium tartarate, etc..[27-30] EDTA and sodium potassium tartrate have been used extensively in traditional electroless copper baths containing formaldehyde as a reducing agent. These plating baths produce low plating rates due to the formation of the cupric complexes and the shift in reduction potential toward negative values. Moreover, EDTA is non-biodegradable and produces serious waste disposal problems.[31] In last few decades, the use of Triethanolamine (TEA) has been used to yield higher plating rates compared to when using EDTA.[32] However, high plating rates are associated with poor bath stability and deposit quality. Tartrate based chelates are used for low plating rate at low temperature. Tartrates are easy to dispose during waste treatment but are not suitable for high speed plating processes. Other biodegradable chelating agents are polyols. Polyols easily form chelates with Cu(II) ions in alkaline medium. The examples include glycerol, xylitol, saccharose, alditol, erythritol, adonitol, D-mannitol, D-sorbitol, maltitol, lacticol etc., for eco-friendly chelating agents for alkaline electroless plating.[33]

3.4. Reducing Agents

The role of reducing in electroless plating is most important as it reduces the cupric ions (Cu^{2+}) to metal atoms copper (Cu) without any change to cuprous oxide (Cu+). Traditional electroless copper plating baths often use formaldehyde as the reducing agent.[13] While using formaldehyde, the electroless copper deposition can be represented as:

$$Cu^{2+} + 2HCHO- + 4OH- \\ \rightarrow Cu + 2HCOO- +2H_2O +H_2 \quad (4)$$

However, formaldehyde is not completely effective in alkaline pH values due to oxidation of the plating bath.[14] In addition, formaldehyde is volatile and toxic in nature. Non-formaldehyde reducing agents used currently include, glyoxylic acid], hypophosphite], sodium bisulfate ($NaHSO_3$), sodium thiosulfate pentahydrate ($Na_2S_2O_3,5H_2O$), Co(II),[46] Fe(II) etc.[16-22,27-32] Sodium hypophosphite is the most popular reducing agent used in copper electroless bath due to its low price, bath stability, and relatively easy to control plating conditions.[34] The electroless copper plating reaction using hypophosphite is given:

$$Cu^{2+} + 2H_2PO^{2-} + 2\ OH- \rightarrow Cu+ 2H_2PO_3- + H_2 \quad (5)$$

The catalytic activity of hypophosphite is weaker and therefore the substrate must be prior activated with Pd or Ni ions.[17,19]

Glyoxylic acid as an alternative reducing agent for electroless copper plating has been used by various researchers. Glyoxylic acid provides higher plating rates and bath stability compared to that with formaldehyde reductant.[14,15,35] The overall reaction with glyoxylic acid is:

$$Cu^{2+} + 2CHOCOOH + 4OH- \\ \rightarrow Cu + 2HC_2O_4^{-} + 2H_2O +H_2 \uparrow \quad (6)$$

$$CHOCOOH + 3OH- \\ \rightarrow HC_2O_4^{-} + 2H_2O + 2e- + 1.01V/SHE \quad (7)$$

This reaction is commonly accompanied by the Cannizzaro reaction,

$$2CHOCOOH + 2OH- \\ \rightarrow C_2O_4^{2-} + HOCH_2COOH + H_2O \quad (8)$$

Other reducing agents investigated in electroless deposition of copper are Dimethylamine borane (DMAB), hydrazine, aminoborane and phenylhydrazine, but their applications are limited due to bath stability and deposit quality optimizations.[22,36-38]

3.5. Additives

The additives are generally used to prevent decomposition of electroless baths. They behave as bath stabilizers. Additives also affects the physical and mechanical properties of the deposit.[27,38-40] There are various kinds of additives in electroless plating:

(a) Inhibitors: They are used to increase the throwing power into holes and recess. e.g., polyethers or polyoxyethers.

(ii) Levelers: Levelers improve plating thickness uniformity at corners and projections and levels the plated layer, e.g., amines, amide surfactants.

(iii) Brighteners: Brighteners control deposit brightness and hardness. They attach to the copper metal ions during plating and facilitate charge transfer at the electrode. Brighteners accelerate plating rates and also control grain structure and deposit characteristics, e.g., Sulphur containing compounds.

(iv) Wetting agents: They decrease surface tension in solution. Surfactants lower the surface tension of the plating solution and thus, allow better wetting of the electrolyte at the electrode.

The common additives in copper electroless plating baths include thiourea, pyridine, cytosine, glycine, guanine, adenine, guanine, ammonia, sodium dodecyl sulphate (SDS), polyethylene glycol (PEG), mercapto group compounds, benzo triazole (BTA), di-pyridyl etc. The additives are beneficial in modifying crystal size, shape and orientation, and adherent copper deposits.[1,8-10,13-23)

3.6. Bath pH

Bath pH is an important parameter in electroless deposition. Bath pH controls the plating rate, microstructure, surface roughness and the crystallinity of the coating.[41) Whenever there is an oxidation of the reducing agents, it indicates the formation of hydrogen or hydroxyl ions (OH^-). The bath pH should be stable for better efficiency of the process. A change in pH severely affects the deposition rate during plating and hence the mechanical properties can get affected. To overcome unstable bath pH values, various stabilizers such as NaOH, KOH, carboxylic acids and amines are used in alkaline solutions.

4. Applications of Copper Electroless Plating

Last few decades have seen enormous growth and demand of electroless copper plating in microelectronics packaging, aerospace, automotives industries, etc. In advanced 3D packaging technology, copper electroless plating is the material of choice. Recently, electroless copper has been tried for the functionalization of nanostructured materials.[13,14) Production of copper nano particles using hydrazine as reducing agent that have also been tried. Not only metallic but also ceramics and polymers have been coated with electroless copper in various engineering applications.[13) Copper plating on polyamides, acrylonitrile butadiene styrene, polyethylene terephthalate, polypropene, teflon, films are flexible and used in modern flexible stretchable electronics, PCBs and shielding applications.[18)

Lightweight composites using the electroless copper plating method. The pollen grains of the lightweight flowers have been coated with copper electroless coating using Pd catalyst.[42,43) Substrates in solder joints for electronics, multilayer boards via plated through-hole technique is performed using electroless copper plating.[44) Copper plated ceramics are employed in microwave circuits in radar, telecommunication and in spacecraft. Electroless plating is important for various processes in electronic, computer, and metallurgy industry of today.[45,46)

5. Conclusion

The process of electroless copper coating on a substrate is an autocatalytic reduction process. Copper electroless plating involves a chemical bath composed of complexing agents, reducing agents like hypophosphite and various organic additives for better surface finish. The electroless copper process has been successfully applied to various surface protection, decorative, electronics, computers, information technology, telecommunications and satellites. Electroless copper applied on non-conducting base are used in wide range of applications in modern flexible electronic devices and sensors. It is concluded that a good fundamental background of copper electroless plating is needed to understand the various roles of organic components in electroless plating baths so that the properties of copper coatings can be further improved. This will set a new direction for electroless plating in the modern research community.

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