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Investigation of Trivalent Cream Plating Problems

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ABSTRACT

Graphite anodes are used in cream plating of trivial baths. During plating, the trivalent cream is oxidized to the hexavalent cream on the anodes and as a result the cathodic efficiency is reduced, so in order to prevent oxidation, the solution around the anode is separated from the solution around the cathode with a ceramic wall. Another way to prevent oxidation of trivalent cream is to add carboxylic acid. Carboxylic acid reduces hexavalent cream to trivalent cream. Zabotin and Kozlovsky have shown that in a sealed container under a neutral atmosphere, divalent cream ions accumulate in the bath, depending on the pH, temperature, and current density of 30 to 60 percent of the total amount of cream. Macho believes that in trivalent cream plating baths with large amounts of organic matter, the deposition of the cream is independent of the amount of its divalent ions. In this case, the only reaction that occurs is the direct reduction of trivalent worm ions to the metal. With increasing the amount of cream, the reduction of trivalent cream to bivalent is accelerated and with increasing the temperature of the bath, the concentration of organic matter and the density of the trivalent cream reduction process decreases to divalent. The cream can be plated in certain conditions of its trivalent baths.

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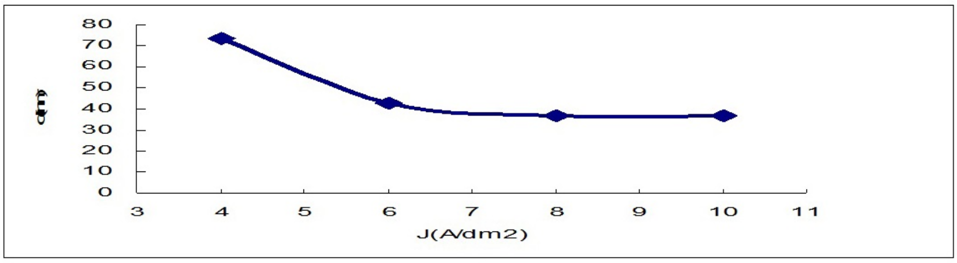
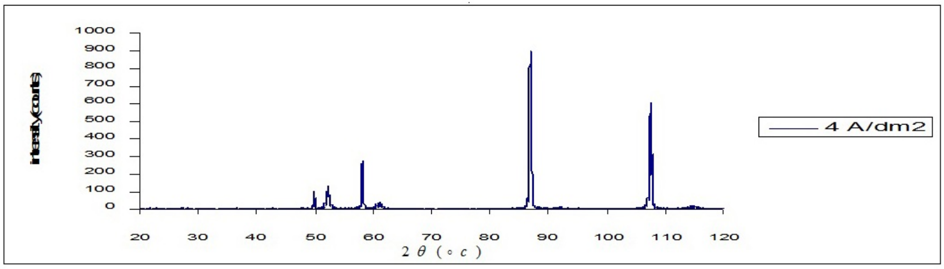
GRAPHICAL ABSTRACT





Introduction

The composition of the bath and the plating conditions should be such that the acidity of the layer adjacent to the cathode is within a certain range. This means that the pH should not be lower than the amount of hydrogen bubbles that are released, as this will reduce efficiency. On the other hand, the pH value should not exceed the amount of cream hydroxide in dilute solution and the precipitation of cream alkaline salts in concentrated solutions. The pH of hydrate formation depends on the amount of soluble cream [1-4]. To prevent the formation of hydrates and alkaline salts, it is recommended to add substances that increase the buffering properties or cause the cream complex. Complexing materials increase the pH of hydrate formation to a greater extent. Much research has been done on how suspended particles are suspended simultaneously with metal ions, but due to many factors that affect the electrochemical deposition of composites, a theory that can combine Predict the chemical and structure of the composite coating [5-7]. These systems have different variables, which include:

(1) Material, size, shape and concentration of particles in the electrolyte.

(2) Applied current density and type of current (direct or pulsed).

(3) Mass transfer conditions (type and stirring rate of the solution).

(4) Electrolyte temperature.

(5) pH electrolyte.

(6) Chemical composition of electrolyte (surface activator and additive) [8].

Classic gaglim model

In 1972, Guglimi [9] proposed a model that was a major step in elucidating the mechanism of particle co-deposition. His model consisted of two successive absorption processes. In the first stage, which is physical in nature, the particles are poorly absorbed by the cathode surface. Particles that are poorly adsorbed on the cathode surface are in equilibrium with charged particles suspended in solution. In the second stage, which is electrochemical in nature, by reducing the ionic cloud around the particles, the particles are strongly adsorbed on the cathode surface [10-14]. These particles, which are permanently attached to the cathode surface, are buried in the growing metal deposit. The model proposed by Gaglimi, unlike previous models, has the ability to consider both the effect of the concentration of ceramic particles in the bath and also to quantify the velocity of particles in the coating as a function of flow density. Guglimi model is very suitable for expressing the mechanism of particle co-deposition using pulsed flow. Since the first stage is not electrochemical adsorption, it does not involve any charge transfer; instead, the co-precipitation of the particles is affected by the hydrodynamic conditions of the bath. As a result, the first phase of adsorption can only take place during the off time when no charge is transferred, and instead the second phase of adsorption, which is electrochemical in nature, occurs only during the light time [15-17].

Recognition of the α-curve in C, where α is the volume fraction of the particles in the coating and C is the concentration of the particles in the bath, is of laboratory and theoretical importance. For coatings created at different values of current density, the C / α curve in α forms a series of straight lines that all pass through a point on the C axis (x-axis) [18-20].

The accuracy of the Guglielmi model has been proven in various co-precipitation systems such as Al2O3 and SiC, ZrO2 and PTFE nickel-based particles [21-23]. By calculating the amount of K, the concentration of particles that are poorly adsorbed on the cathode surface, lσ and the concentration of particles that are strongly adsorbed, sσ using the Langmuir formula optimized by Guglielmi for electrical co-precipitation systems Is a calculation [24-27]. Assuming that all particles that are strongly adsorbed on the cathode are buried in the coating, the percentage of poorly adsorbed particles can be calculated using. Volume percentages of particles that are poorly adsorbed by poor adsorption And strong adsorption are attached to the cathode surface, Table 2-1 shows for the nickel-silicon-silicon composite coating containing nanometer-sized silicon carbide particles with dimensions of 62 nm created by direct current [28-31].

Table 1. Percentage of particles that are adhered to the cathode surface by weak adsorption and strong adsorption during the formation of nickel-silicon carbide composite coating [32]

C%	**$_{l}\sigma$**	**$_{s}\sigma$**
۰,۱	۱۷/۳	۱/۷۶
0.3	۳۷/۵	۳/۳۶
۰,۵	۴۸/۹	۵/۳۸
۱	۶۳/۵	۶/۷
۱/۵	۷۰/۶	۷/۴۶

It is observed that the concentration of particles that are poorly absorbed by the cathode is much higher than the concentration of particles in solution. In addition, only 0.1%

of particles that are poorly adsorbed on the cathode can enter the coating [33-35].

As a result, it can be said that the stage determining the rate of particle participation in the coating is controlled by the process of converting weak adsorption to strong adsorption of particles [36-38].

Celis model

In 1987, Celis [39-41] proposed a model that, unlike the Guglielmi model, also included the effect of the hydrodynamic flow of the solution. According to this model, the co-precipitation of ceramic particles in a growing coating consists of the following five consecutive stages:

(1) Formation of an ion cloud around the suspended particles in the plating bath [42-45].

(2) 2- Transfer of particles from the hydrodynamic boundary layer by displacement currents in the solution to the cathode.

(3) The passage of particles through the concentration layer by the penetration mechanism.

(4) Passage of particles through the electric double layer by electrophoretic process and surface adsorption of particles to the cathode [46-49].

(5) Strong adsorption of particles on the cathode surface, which is done by reducing the right amount of ions.

in 1992, Fransaer [50-55] and his colleagues, in justifying the Cu-PS and Ni-SiC co-deposition systems, proposed a new model based on the trajectory of non-Brownian particle particles. In this model, the path of the particles is determined based on all the forces and torques that act on the particles, such as the force of gravity, the forces due to electro phoretics and the forces applied by the double layer. Their model consisted of two stages [56-59]. This model was not true near the electrode surface as it would lead to complete immersion. Because of this, they introduced a phrase into the proposed equations that related to the interaction of the particle and the electrode. Thus, they proposed a new equation for determining the probability of a particle entering the coating, which provided a suitable explanation for the changes in the concentration of particles in the coating in terms of the concentration of particles in the solution. This model did not have the ability to predict the maximum concentration of particles in the coating in terms of current density changes [60].

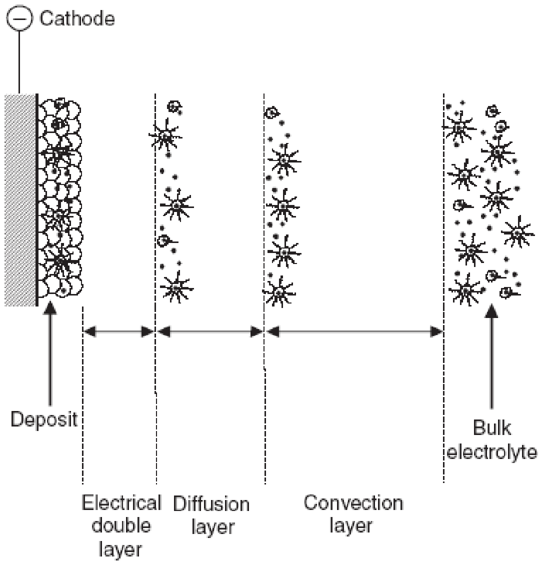


Figure 1. Celis five-step model for metal particle co-deposition

Colloidal stability

In colloidal systems where the dispersed component or colloid has at least one dimension in the nanometer to micrometer range, colloidal stability and colloidal dispersion is a very important issue. Accidental collisions between scattered particles often occur in a liquid medium, and since composite plating electrolytes are examples of colloidal systems, there is a possibility of insoluble particles joining [61]. As a result of the adhesion of these particles in the solution, the diameter of the reinforcing particles from the dimensions of less than 100 nm to the micrometer dimensions, and this causes poor bonding between the particles in a lump, which facilitates crack germination and reduces tensile strength and Leads to ductility and abrasion properties and hardness of coatings [62]. Particles distributed in an electrolyte solution are in constant brown motion. When two particles come together, there are energies between the two particles that determine whether the particles will separate or agglomerate. Particle agglomeration generally occurs as a result of gravity energy greater than repulsion energy between particles. The size of the net forces involved in forming the agglomerated structure clearly depends on the condition and nature of the system [63]. Physical distribution of nanoparticles by ultrasonic operation: In this method, particles are separated from each other using destructive energies such as ultrasonic vibrational energy. The propagation of ultrasonic waves in solution produces very large local pressures thousands of times the atmospheric pressure, which breaks the bond between the particles in the lump and prevents the particles from clumping [64].

Chemical methods of dispersion of colloidal systems

In this method, by adding surfactants, by reducing the ionic strength of the bath and by changing the pH, the diameter of the agglomerates can be reduced.

Changing the pH: Changing the pH of the plating bath changes the zeta potential of the particles. Zeta potential is the electric potential that exists at the boundary between the solid surface and the liquid environment in which the solid particles are located and is a function of the surface charge of the particle, the adsorbed layer at the interface, the type and composition of the medium in which the particle is suspended. The magnitude of the zeta potential of a particle is a measure of the interaction of particles, so that it can be used to predict the long-term stability of a solution. If particles have a positive or negative charge, they tend to repel each other and resist agglomeration. If the particles have low zeta potential, there is no factor to prevent the particles from agglomerating and as a result, the particles agglomerate. The boundary between stable and unstable solutions is +20 mV and -20 mV, which means that if the zeta potential of the solution is more than + 20 mV or less than -20 mV, the solution is usually stable, and if the zeta potential is between 20+ mV and -20 mV are unstable solutions and agglomerated particles [65].

Most substances have zeta potential when suspended in water. Zeta potential is affected by factors such as pH, additive concentration and ionic strength of the solution. The most important factor that affects the zeta potential is pH. The zeta potential of many chemical surfaces, such as mineral oxides in dilute salt solution, show a certain behavior by changing the pH, so that when the pH increases with the addition of alkalis, the zeta potential becomes more negative or at least Its positive value decreases. By adding acidic substances, their ionization reduces the amount of hydroxyl ions and thus the particle surface becomes more positive. In the zeta potential curve in terms of pH, there are pHs where the curve crosses the zero potential. This point is called the isoelectric point. This point indicates the pH value at which the solution has the lowest stability at pH [66]. The use of pH changes to reduce the diameter of the lumps should be done with caution as these changes may cause deterioration of the quality of the coating [67]. Use of surfactants When surfactants or scavengers are immersed in a solution or mixture of emulsion and suspension, they have a strong tendency to form adsorption joints, thus reducing surface tension and wetting and dispersing. Tarsaz is an organic molecule that has a water-soluble part (hydrophilic) and a fat-soluble part (hydrophobic).

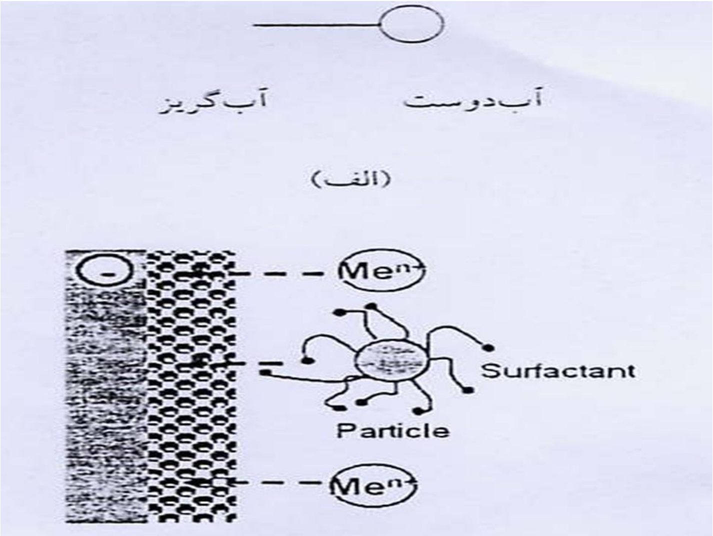


Figure 2. Schematic representation of an activator and the performance of activators in the electrolyte

According to the structure of scarecrows, they are divided into 4 main types: anionic, cationic, non-ionic and amphoteric. In anionic turbulence, the water-soluble part has a negative electric charge, while the cationic types have a positive charge. Non-ionic instruments are chargeless and amphoteric devices have positive and negative charges on each part of the molecule. Wet instruments scatter the particles by increasing the electric potential (electric charge) of the surface. By using scavengers that have electron-rich aromatic rings and a flat molecular structure, by absorbing them much more easily or by creating a barrier of organic matter around the particles, particle aggregation is dramatically avoided and even completely prevented. The transparency of the solution is a critical measure of colloidal stability and is determined by ocular observation. Depending on the concentration of turpentine, different states are seen in the transparency of the solution. At somewhat higher concentrations, although the wetting concentration is still very low, the solution appears cloudy or cloudy. As the concentration of the wet solution increases, the cloud-shaped solution gradually turns into a milky solution, which typically has colloidal stability conditions, and at higher concentrations the solution will become completely clear. Surface activators are not only a dispersant of particles in the electrolyte but also increase co-deposition. However, there are two problems with the use of conventional wet instruments, which are:

(1) Some of the scavengers remain on the fine particles and enter the coating with them and prevent the deposition of other particles, so in this case the amount of deposited particles is limited [67].

(2) The coating and may have an adverse effect such as brittleness in the coating [67]. The effect of a tarsus on simultaneous deposition depends on the following factors:

(1) Particle dispersion ability.

(2) Properties of more ionic instruments.

(3) Interaction of electrodes and particles with triggers. To create a dispersion stability in the composite plating solution, first a more specific amount of solvent is dissolved in the solution, then a certain amount of particles is added to the solution and then the solution is stirred for 5 minutes by ultrasonic waves and finally the solution for 30 minutes before It is mechanically agitated by plating [68].

Impact of plating flow type

Direct currents, square pulses, triangles and reverse pulses can be used to create a composite coating by electroplating method. Although direct current has been used in many cases to create composite coatings with nanoparticles, studies have shown that the use of pulsed current in addition to the possibility of creating a wider range of composite compounds leads to the presence of more nanometer particles in the coating. Pulse flow plating can improve hardness, ductility, surface roughness as well as less porosity and more abrasion resistance. In pulsed current plating, a finer and more uniform structure is available than direct current because it prevents the growth of crystalline plates during blackouts and at the same time encourages light at the beginning of the nucleation process [69].

During the pulse, the pH and the concentration of metal ions on the cathode can be recovered at the time of extinction. The recovery of metal ions and hydrogen depends to some extent on current density, frequency and duty cycle [70]. The pulse reverse current has the same characteristics as the pc current, except that the cathode current is established during the illumination time and the anodic current during the off time. This method is known as a successful method for displaying higher concentrations of nanometer particles [71]. Using reverse pulse current, three major advantages can be achieved compared to direct current.

(1) More nanometer particles can be achieved in the coating.

(2) Lower concentration of nanometer particles in solution can be used.

(3) Selective presence of smaller particles in the coating is achievable. All three are due to the partial dissolution of the deposited metal during anodic flow, so that larger particles tend to separate from the coating while smaller particles remain in the coating [72].

Impact of lighting time and off

The results of recent studies on nano composite coatings produced by electroplating show that the length of extinguishing and light times as well as their ratio have a great effect on the crystallographic texture and morphology of the background as well as the amount and distribution of ceramic particles in the background. Increasing the lighting time in the range of 1 to 5 milliseconds leads to a decrease in grain size, which is due to the increase in potential and, consequently, the increase in nucleation speed. Also, increasing the lighting time leads to a change in the back ground texture from a random distribution to a strong fibrous texture (200). On the other hand, longer extinction times cause grain size to enlarge and change the crystallographic orientation. Free ions reach the cathode surface faster than ions on the surface of ceramic particles due to their greater mobility. As a result, more free ions are reduced during the luminosity than ions at the particle surface. During the shutdown time, because charged particles do not compete with free ions to reach the cathode surface. Particles have a better chance of reaching the cathode surface, which increases poor particle adsorption. As a result, with increasing extinguishing time, more particles enter the coating and this is in agreement with the results of many researchers. However, increasing the illumination time can increase the probability of particles being removed from the cathode surface, which reduces the presence of particles in the coating.

Influence of current density

Since it has been proven today that nanostructured materials have different properties from micro-structures, researchers are trying to produce nanostructures and study their properties. In electrochemical coating method, by controlling electrochemical parameters, nanostructured coatings can be achieved. One of the important parameters that plays an important role in grain size granulation is current density. X-ray scattering pattern obtained from nickel nano crystalline coatings by applying. The grain size of nickel nano crystals is obtained from the X-ray scattering pattern (XRD) using the Scherer discharge relation.

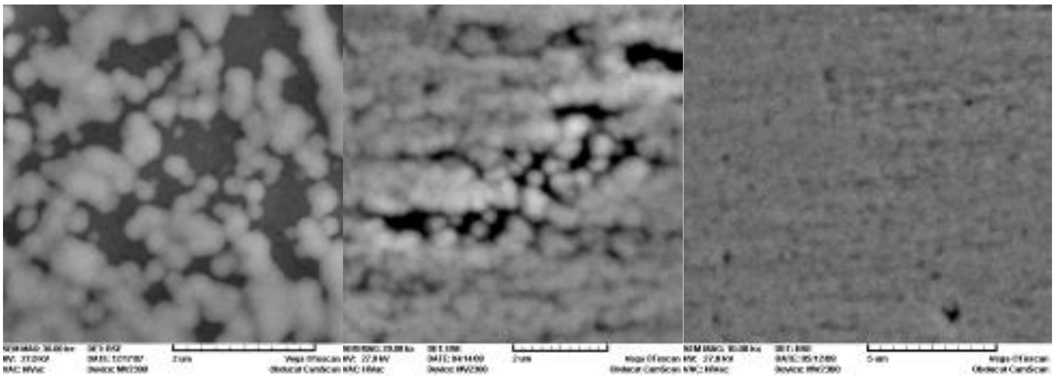


Figure 3. Nano-nickel coating by applying current density a) 4, b) 6, c) 8, amperes per square decimeter

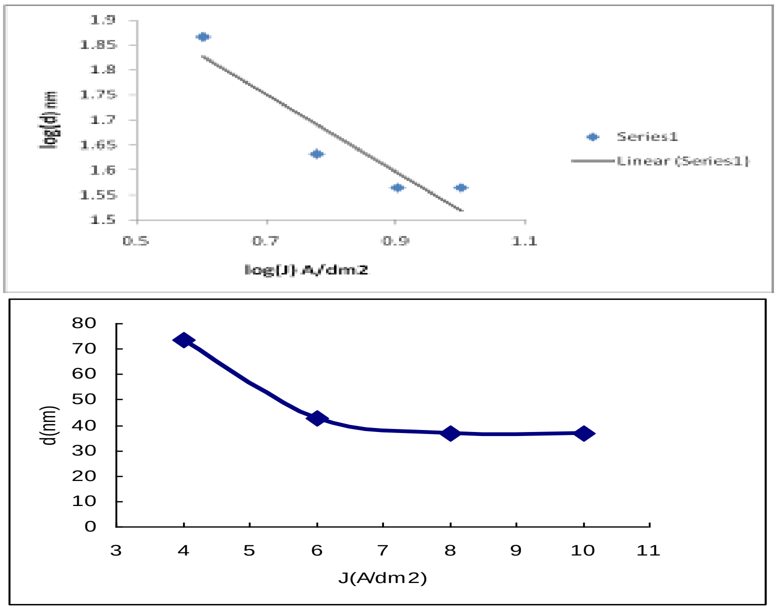


Figure 4. Relationship between coating current density and grain size of nickel nano coating

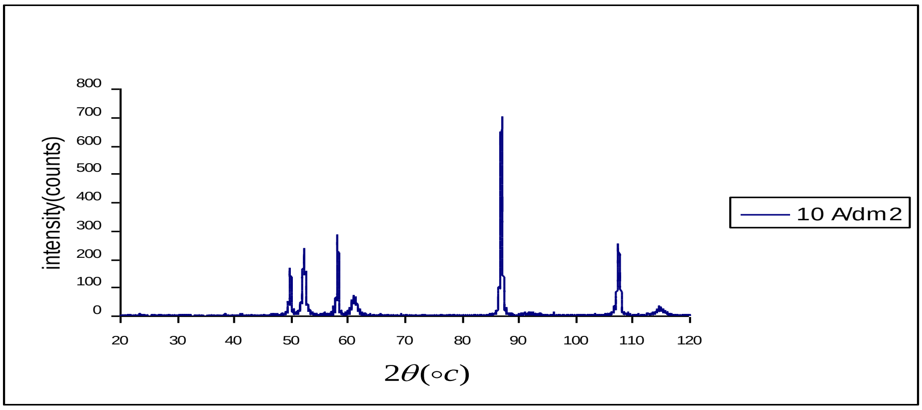


Figure 5. X-ray diffraction diffraction pattern of nano-nickel coating in different current densities

Conclusion

Some researchers have reported that an increase in current density in the coating of nickel nanoparticles from a sulfa mate bath has resulted in fine-graining, which has been reported to increase germination rates. While Cziraki reports that an increase in current density in the nickel coating of the sulfate bath has increased the grain size of nickel crystals, what other researchers have shown is that in different baths, the effect of current density on nickel nano crystal finesse It is different. At high current densities, with increasing current density, there is no significant increase in micro granulation, which can be related to two phenomena:

(1) At high current densities, the growth rate of nickel clusters is controlled by mass transfer and hydrogen evolution at the cathode surface.

(2) Inactivation of the cathode surface by the precipitation of nickel hydroxide causes the application of higher current density to increase the passive surface.

Also, the current density has a great effect on the amount of ceramic particles introduced in the nano composite coatings produced by electroplating method. In another study on 32 nm alumina particles, it was found that the type of electrolyte affects the presence of ceramic particles in the coating, so that in chloride electrolyte the presence of more nanoparticles in low current density is observed while in citrate electrolyte in high current density higher concentration Particles were obtained in the coating. This shows that the effect of current density changes with changing electrolyte composition.

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