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Development of alternative environment-friendly nickel and gold plating baths

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Abstract

In the metal plating industry, nickel and gold plating baths are used, which contain toxic materials; moreover, they cause important wastewater problems. Despite these disadvantages, there is a strong need for both these types of metal plating because of their aesthetic properties. However, until now, nickel plating could not be used with gold plating because nickel plating baths contain sulfates. In this study, gold plating on nickel plating that contains acetylacetonate salts is investigated. The physical parameters of the plated materials, which were obtained with good results, were determined by hydrogen embrittlement tests, visible region spectrophotometry, and the Charpy impact test.

Keywords: gold cyanide, nickel(II) acetylacetonate, dimethylgold(III) acetylacetonate, embrittlement

1. Introduction

1.1 Nickel plating

Metal plating, which is a treatment adopted to improve the surface properties of metals, is being widely used in surface plating applications. Nickel is the most commonly preferred plating metal from the viewpoints of corrosion prevention and aesthetic value [1]. With the development of nickel plating baths, considerable improvements have been made to the metal plating process. To understand the corrosion resistance of a nickel-plated surface, a salt spray test is performed using 5% NaCl at 37 °C and 100% relative humidity. Various bath compositions have been proposed on the basis of the ASTM B117 standard for use in nickel plating [2]. The composition of the nickel plating bath can be modified as per the required features of the plated surface. Among the different types of nickel plating baths available, the Watts-type bath is most often preferred [3] because many of the bath parameters can be easily controlled [4]. In the last few years, many research groups have performed studies on alternatives to traditional nickel baths. Buffer solutions such as sodium acetate, acetic acid, citric acid, and citrate been used to control the parameters pertaining to nickel plating baths [5]. However, the buffer solutions used for electroless plating baths differ from those used for electroplating baths [6]. Studies have been performed on the feasibility of using complex salts such as iron alloys [7], nickel–tungsten alloys [8], and copper–nickel alloys [9] in buffer solutions for electroless baths. There are numerous methods for increasing the aesthetic value of nickel-plated surfaces. One of these involves the use of a brightening agent. Some examples of such brightening agents include Ni-S-C alloy [10], Ni-Ru-Al alloy [11], and Ni-Al alloy [12]. TiN plating on mild steel was attempted by Subramanian [13]. Ni-P plating on W-Cu was performed by Wang *et al.* [14]. Another example of Ni-P plating is that performed on a microarc oxidized magnesium alloy [15]. The use of a Ni-Fe-P alloy system for nickel plating was studied by Yung *et al.* [16]. Further work on the corrosion resistance of Ni–P

plating was carried out by Rahimi *et al.* [17]. Nickel plating characteristics on AISI were studied by Abdeli *et al.* [18]. Ni-P plating on W-Cu alloy was carried out by Hao [19]. Nickel plating on Ni-B alloy was performed by Srinivasan, who obtained good results [20]. Effect of the nickel plating on metal surface were studied with ZrO_2 particles by Arghavanian [21]. On the other side, nanocomposite plating of Nickel plating was important performed study by Hasannejad [22].very good results were obtained in performed studies with different composites in industry[23].The properties of the nickel plating may vary considerably depending on the bath composition, plating time, and temperature. The characteristics of nickel plating on Colmonoy, a nickel alloy, were investigated by Ramachandran *et al*. [24]. The use of tartrate solution in the nickel plating bath was studied by Hosseini *et al.* to improve the features of the plating [25]. Ni–P–Co plating of a nanoalloy was studied by Srinivasan *et al*. [26]. The use of a Ti-Ni composite to improve the strength of the plating layer was studied by Zanganeh –Medar *et al*. [27]. A nickel–ceramic composite membrane was used by Bulasara *et al.* to evaluate bath performance in Ni plating [28]. Another study on the TiN-Ni-MiS system was performed by Subramanian *et al.* [29]. Gezerman *et al.* [30] identified an alternative to conventional nickel baths and studied the effect of dimethyl(acetylacetonate)gold(III) on nickel plating [31]. The current study differs from the previous ones in terms of the scanning electron microscopy (SEM) parameters and the type of brightening agent used. In the present work, good plating properties were achieved for nickel and gold even with a small amount of brightening agent.

In addition to these factors, because of the toxic properties of nickel and gold plating baths, a different composition from those used in published works is studied here, and better hydrogen embrittlement of plated materials is obtained. Further, hydrogen cracks on -these materials are observed by electron microscopy. In the metal plating industry, it is desirable to prevent

problems associated with wastewater and use the minimum possible amount of chemicals; the overall aim is to minimize the cost of the plating baths, which has been achieved herein.

As mentioned above, Watts-type baths, which have similar inorganic compositions, are generally used for nickel plating [6,8]. All the plating experiments in this study were performed using a Hull cell [32] at 50 °C without stirring.

1.2 Gold plating

Gold plating of metals has a number of applications arising from the chemical and physical properties of gold, such as its resistance to corrosion and high electrical conductivity. As a result of its aesthetic value, gold plating is also widely used for decorative purposes. Ii~~i~~nterlayers of different materials were used by Santos *et al*. to improve the corrosion resistance of gold plating and achieved good results [33]. Tetrachloroaurate(III) solutions containing hexacyanoferrate(II) ions were used by Vrublevskaya *et al*. for gold plating [34]. Decorative soft gold plating was studied by Cavallotti *et al.* [35]. One non-electrolytic approach to the deposition of gold ions from an aqueous solution involves ultrasound treatment of tetrachloroauric(III) acid; for example, this sonochemical reduction was used by Zhang *et al*. to create gold nanoparticles [36]. Electrolytic plating of gold on an AA100 aluminum alloy was investigated by Mohan and Raj [37], whereas the use of gold plating in the fabrication of optoelectronic devices was studied by Roy and Caprodossi [38]. As with nickel plating, there are environmental concerns regarding the materials used in the gold plating process. An important study on the toxicology of gold plating baths was conducted by Roczanski *et al.* [39] who found that, in particular, extremely toxic cyanide salts are widely used in gold plating, and therefore, there is a strong incentive to look for alternatives such as dimethylgold(III) acetylacetonate that have minimal environmental load .

1.3 Gold plating on nickel plating

It is difficult to coat a previously plated metal surface with another material by electroplating,

at least with the existing techniques. In order to overcome this difficulty, it is necessary to modify these techniques or to develop new approaches. One problem with gold plating on a previously nickel-plated surface is the presence of a passive nickel(II) sulfate layer arising from the use of nickel(II) sulfate salt in the plating bath [40]. This problem can be solved by replacing the sulfate salt with nickel(II) acetylacetonate.

The main subject of the present study is gold plating on a nickel-plated surface. The effects of nickel(II) acetylacetonate in a nickel plating bath on the amount and thickness of the nickel deposit and its physical properties were measured by electron microscopy and visible-light spectrophotometry. The results of this study may also be useful in accelerating the adoption of a mixture of sodium acetate and acetic acid as a substitute for boric acid as a pH buffer. Moreover, the use of dimethylgold(III) acetylacetonate for gold plating on nickel plating was investigated.

2. Experimental procedures

2.1 Materials

The main composition of the nickel plating bath used in the experiments is shown in Table 1, and the analytical results for the bath are shown in Table 2. The composition of the carrier is shown in Table 3 and that of the brightening agent is shown in Table 4. The materials used for the analysis of the nickel content of the bath were 25% ammonia solution (NH_3/H_2O, pH 11.2), murexide indicator, and 0.15 $mol \cdot L^{-1}$ of ethylenediaminetetraacetic acid (EDTA). All the materials were reagent grade, obtained from Merck Chemicals (Istanbul, Turkey), and used without further purification. The analytical results for the gold plating bath are shown in Table 5, and the main composition of the bath is shown in Table 6. Sulfuric acid (98%) was used to analyze the gold content of the bath.

2.2 Plating experiments

2.2.1 Nickel plating

As a preliminary step, 70 cm^2 (10 cm × 7 cm) of shiny AISI 304 stainless-steel plates were exposed to a current of 1 A (0.1 $A \cdot cm^{-2}$) for 10 min in the bath. However, because the coating obtained in this first step was not sufficiently thick, the plates were subjected to a second bath with the same chemical composition but under increased current density (0.3 $A \cdot cm^{-2}$) for 10 min. Before plating, the plates were cleaned in a degreasing electrolytic bath and rinsed with tap water. Then, they were neutralized in a 20% sulfuric acid solution and rinsed once again with tap water. The plates were used in the experiments without being dried. The Hull cell tests were carried out with varying concentrations of the carrier and brightening agent. The quantities of the brightening agent and other compounds were determined in separate experiments. To ensure a standard concentration of the various compounds, a large volume of the mixture was used. Thus, the proportions were kept constant, irrespective of the quantity that was added to the bath.

2.2.2 Gold plating

As with nickel plating, the working performance of the gold plating bath was also investigated using a Hull cell. The current densities were 1 A (0.1 $A \cdot cm^{-2}$) and 4 A (0.4 $A \cdot cm^{-2}$) for 15 s, with different plating thicknesses being observed for different current densities. After the Hull cell tests, the gold-plated samples were first immersed in 1% sulfuric acid and then in tap water. The described gold plating process is flash electrolytic plating, which results in low plating thicknesses. The anodes used in the gold plating process can be stainless steel and zinc, both of which aid in the control of aesthetic properties such as brightness.

2.3 Plating bath analyses

2.3.1 Nickel analysis

1 g sample of the sample was transferred from the nickel plating bath to an Erlenmeyer flask, and 100 g of pure water, 10 g of aqueous ammonia (25%, pH 11.2), and a small quantity of murexide indicator were added. This mixture was titrated with EDTA until a color change

was observed. The nickel (Ni^{2+}) content in grams per liter was determined by multiplying the amount consumed by a factor of 5.87.

2.3.2 Gold analysis

A 50 mL sample of the gold plating bath was added to a 1 L Erlenmeyer flask, to which was added 25 mL of 98.6% H_2SO_4. This solution was heated until precipitation of gold occurred (approximately 1 h). The precipitated gold was washed with warm water, placed in a crucible, and heated until red hot. After cooling, the resulting gold foam was weighed. Multiplication of this value by 20 revealed that 1 L of the plating bath contained 4.2 g of gold.

2.4 Electron microscopy

A Zeiss Supra 55 scanning electron microscope was used in the secondary electron image mode, with the following parameters: resolution, 100 µm; high-voltage separation, 10.0 kV; working distance, 6.8 mm; and magnification, ×100. A total of six samples from six baths were examined: three with differing proportions of the nickel-brightening agent, and three with differing proportions of the brightening agent and gold plating.

2.5 Color analysis

The effects of the brightening agent containing complex organic salts and those of gold plating on the color of the metal plate were examined in the CIE (Commission Internationale de l'Éclairage, translated as the International Commission on Illumination) *L*a*b* color space [41-43] using a visible-region spectrophotometer (Macbeth CMC-CE-7000-XL0339) at 325 nm and CIE Standard Illuminant D65, which is equivalent to 6500 K illumination. These values are in accordance with European and Japanese standards. Two measurements were taken from each plate surface. Our aim was to perform highly sensitive color measurements in the visible color region and to supply the values closest to market requirements. Nowadays, visible-color spectrophotometry is not used for color analysis in the metal plating industry; however, in this study, because of the subtle differences in the color of the gold plating on

nickel plating, visible-color spectrophotometry was used. Comparisons of color values are more recognizable with this system, and this method is used to measure the contrast between a certain specified standard color and a sample color. In the CIE L^*a^*b color system, the L^*, a^*, b^*, c^*, and h^0 values are the main parameters used in defining the colors. The definitions are given below:

L^*: the central vertical axis. The values used are 0° for black and 100° for white. The L values range between these two values. The L^* value represents lightness.

a^* and b^*: chromatic coordinates in the CIE L^*a^*b color space, representing $+a^*$ red, $-a^*$ green, $+b^*$ yellow, and $-b^*$ blue.

c^*: the chromaticity. The chromaticity value of a color increases as the value drifts horizontally from the center of the color space. It gives information about the saturation of the color. A color and its tone at the same level are the characteristics defining the distinction level from another color (between black and white). If a color diverges from black, its saturation will increase; if it approaches gray, its saturation will decrease. The exact gray saturation is 0. Because each color's maximum saturation degree varies, the upper limit cannot be indicated. In the CIE L^*a^*b color space, the c^* value is measured by the angle between the measured color's projection in the axis of ordinate and its angle in the axis of ordinate.

h^0 (hue): the basic color of an object (such as red, blue, yellow); it is the angle between c^* (chromaticity) and the axis of the abscissa. Angle h^0, which indicates the color intensity, is 0° and 360° along $+a^*$, 90° for $+b^*$, 180° for $-a^*$, and 270° for $-b^*$. The h^0 value can be defined as the angle between the axis of the abscissa of a measured color and the axis of the abscissa of a standard color [44].

2.6 Mechanical hydrogen embrittlement test

A progressive power test method was used to measure the hydrogen embrittlement of the samples. A force of 14.701 N was applied to a plate moving progressively between two other plates, as per the ASTM F519-10 standard [45].

2.7 Thickness measurement

X-ray equipment (Fischer Instruments GmbH, 2002) was used to measure the plating thickness and thus obtain accurate information on the Ni and Au percentages, as well as the average, maximum, and minimum values and the standard deviation.

2.8 Charpy impact test

The Charpy impact test was conducted using a ZBC 2000 Pendulum Impact Testing Machine (Sans, China). As per the ASTM E23 standard, a force of 3 $kg \cdot cm^{-2}$ with a fall speed of 5.5 $m \cdot s^{-1}$ was applied to the metal surface. Adhesion forces were measured in the high-current-density regime, in which the nickel coating thickness was 10–15 μm and the gold coating thickness was 0.25–0.50 μm; and in the low-current-density regime, in which the nickel coating thickness was 1–5 μm and the gold coating thickness was 0.010–0.050 μm.

3. Results and discussion

The results showed that the thickness of the coating and its adhesion to the metal substrate surface increased from 1 μm to 15 μm and from 0 $kg \cdot cm^{-2}$ to 3 $kg \cdot cm^{-2}$, respectively, with increasing current density [45]. In the low-current-density regime, the thickness of the nickel coating was less than 10 μm and that of the gold coating was less than 0.25 μm; the coatings separated from the substrate surface on impact in the Charpy test. However, in the high-current-density regime, the coating was thicker and showed strong adhesion to the substrate surface; in other words, the coating did not peel off the surface (Table 7). For nickel plating in a Watts-type bath, the current density should generally be between 0.1 $A \cdot cm^{-2}$ and 0.5 $A \cdot cm^{-2}$ in order to ensure uniform coating and optimized chemical consumption [46]. In the present experiments, a current density of 0.3 $A \cdot cm^{-2}$

resulted in uniform plating on the metal substrate (Table 8). From the electron microscope images (Figs. 1–3), it is apparent that the amount of brightening agent used in the nickel plating bath influenced the brightness of the nickel plating and that of the gold plated on nickel. Further, the gold plating on the nickel plating appeared to be brighter than the gold plating formed directly on the substrate. The effects of brightening agents on the nickel and gold plating were also investigated using a visible-region spectrophotometer. When different concentrations of brightener were used, different shades of plating were observed on the plated materials. To determine the optimum concentration of the brightening agent, it is also necessary to consider the effects of pH. An increase in the amount of brightening agent added to the bath leads to an increase in the pH of the bath [8], which in turn increases the hydrogen embrittlement of the plating. In this experiment, the deleterious effects of hydrogen on the tensile properties of metals are caused by the association and movement of hydrogen within the dislocations. Hydrogen–dislocation interactions modify metallic deformation processes by stabilizing the microcracks, changing the work-hardening rate, and inducing solid-solution hardening (Figs. 4–6). The extent to which such modifications cause embrittlement depends on the properties and defect structures of the uncharged steel, as well as on the hydrogen-induced changes in the deformation processes [47].When metals are plated, cations and hydrogen ions in the buffer solution play an important role in cohesion to the metal surface. Thus, a decreasing hydrogen ion concentration (increasing pH) results in weaker adhesion and increased brittleness [48]. At pH 5.0, cracking occurred 11 h after the test; at pH 5.5, cracking occurred 10 h after the test; and at pH 6.0, cracking occurred 9 h after the test. The desired brightness effect was obtained at a brightening agent concentration of 0.6 $g \cdot L^{-1}$, but a further increase in concentration increased the degree of hydrogen embrittlement (Figs. 4–6).

The concentration of carriers in the nickel plating solution did not affect the nickel coating, according to the results of the SEM analyses. Under D65 illumination (Figs. 7–9;

Tables 9–12), the brightening agent plates located on the light side of the light–dark axis exhibited an increase in brightness with increasing brightening agent concentration. In contrast, upon the addition of the carrier, the color and brightness did not diverge from the standard (original) bath plate axis. The hue h^0 and chromaticity c^* of the samples did not change significantly at the point of saturation or from the yellow hue (because of the gold plating). Similarly, L, which determines the lightness/darkness ratio, did not change upon the addition of either agent. Note that in the measurement of L, the distance to the standard rectangle located at the middle of the light–dark scale was used as reference. To determine the optimum quantity of carrier, we started with 8 $g \cdot L^{-1}$ of carrier and then increased the amount by 25% to 10 $g \cdot L^{-1}$, which gave the plating a matte texture. Changing the concentration of carrier did not affect the pH (Table 13) or hydrogen embrittlement. In the experiments on plating gold onto a previously nickel-plated surface, dimethylgold (III) acetylacetonate was used instead of cyanide salts. The required physical properties of the gold coating, i.e., brightness and lightness, increased with increasing amounts of dimethylgold (III) acetylacetonate (Figs. 10–12). Increasing the amount of dimethylgold (III) acetylacetonate did not decrease the pH further, and therefore, no change was observed in the embrittlement of the plated material. The nickel sulfate solution used in traditional nickel-plating baths results in cohesion problems when gold plating is applied on top of the nickel plating. Therefore, nickel (II) acetylacetonate solution was used, which prevented the growth of a passive sulfate layer on the nickel plating. In the Charpy impact test, coating thickness measurements were performed, which indicated that the thickness of the gold plating was unchanged and that the plating was stable with good adhesion properties, i.e., it did not peel off the surface. Moreover, the economical bath compositions described herein help in achieving improved corrosion resistance in both gold and nickel plating and allow for adequate control of aesthetic features (such as bright versus matte), depending on the kind of anode adopted. Depending on

the industrial sector in which the metal plating will be used and the customer requirements, gold can be electrolessly plated on a nickel-plated surface. However, the most important problems associated with electroless plating baths are poor cohesion on the metal surface and low corrosion resistance. With the compositions suggested in this study, electroless plating of nickel and gold was achieved with an electrolytic plating bath. In this manner, depending on the desired corrosion resistance, aesthetic properties, and market requirements, different plating effects on metal -surfaces can be achieved.

4. Conclusions

In this study, a new composition that enables the combined use of nickel and gold plating is chosen. These metal platings are preferred in the metal industry because of the decorative and anti-corrosive properties of nickel and gold plated materials. This is a very important development to enable the industry to minimize wastewater problems.

Dimethylgold(III) acetylacetonate did not affect the color of the gold plating in terms of its decorative properties; it is appropriate to use a dimethylgold(III) acetylacetonate concentration of 1.5 $g \cdot L^{-1}$ at 0.4 $A \cdot cm^{-2}$ to give a plating thickness of 0.25 µm.

In this study, determination of hydrogen embrittlement, physical parameters like plating thickness, and the color effects for nickel and gold platings were also conducted. The presence of a carrier in the nickel plating bath accelerated the deposition of nickel ions and the cohesion ability on the metal cathode surface, but it did not affect the pH of the bath. The use of a brightening agent for the nickel plating bath decreased the current density but increased the pH of the plating bath.

In studies performed to determine the optimum concentration of brightener and carrier agents used in the nickel plating bath, the brightener concentration was determined to be 0.6 $g \cdot L^{-1}$, and the carrier concentration was determined to be10 $g \cdot L^{-1}$. The operating parameters for nickel plating were a temperature of 50 °C, a current density of 0.3 $A \cdot cm^{-2}$,

and a deposition speed of 0.4 $\mu m \cdot min^{-1}$. The operating parameters for gold plating were a temperature of 30 °C, a current density of 0.3 $A \cdot cm^{-2}$, and a deposition speed of 0.1 $\mu m \cdot min^{-1}$.

In addition to clarifying the mechanical and physical properties of the plated material, the plating studies also help in minimizing wastewater problems, as the number of chemical agents- used is minimized. As a result, process costs are reduced, so that the proposed technique is commonly preferred by manufacturers.

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