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Using Taguchi Experimental Design Method for Obtaining Suitable Thickness in Decorative Chromium Electroplating

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Abstract

In this study, Taguchi experimental design has been used for the optimization of thickness of chromium layers. For this purpose brass materials have been electroplated under various conditions of the process. An orthogonal array (OA) was employed to analyze the effect of plating parameters on the characteristic of the thickness. Variables which were considered for working were as following; amount of chromic acid, amount of sulfuric acid, time, and current density. Eventually, the obtained results revealed that among the influential parameters in the process, only current density of 6A/dm^2 and time of 4 minutes have the most effect on the enhancement of the thickness.

Keywords: Thickness; Electroplating; Decorative Chromium; Taguchi Method; Experimental Design

1. Introduction

Chromium electroplating process is the most effective way of protecting the materials against irreconcilable effects of environment or improving surface features of the base material. Hence, this process is widely used in many plastic molds and mechanical parts due to its good aesthetic appearance, good mechanical property and superior corrosion resistance [1-2]. The electroplating is an electrodeposition process for producing a dense, uniform, and adherent coating, usually of metal or alloys, upon a surface by the act of electric current [3]. The coating produced is usually for decorative, protective purposes, and enhancing specific properties of the surface. The surface can be conductor, such as metal or nonconductor, such as plastics. The core part of the electroplating process is the electrolytic cell (electroplating unit). In the electrolytic cell a current is passed through a bath containing electrolyte, the anode, and the cathode.

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Drawbacks of chromium electroplating processes are the use of environmentally unfriendly chemicals such as hexavalent chromium. On the other hand, it is well known that chromium plating is an operation that has serious disadvantages from the environmental point of view [4]. Chromium electroplating operation can be categorized based upon the thickness of the chromium metal layer applied. It can be classified as "hard chromium" and "decorative chromium" electroplating [5]. Decorative protective coatings are primarily used for adding an attractive appearance to some protective qualities. In decorative plating the chromium is plated as a thin layer over nickel to provide an economical and highly corrosion resistant deposit. In this plating application, a thin chromium layer protects the nickel from oxidation and tarnish while providing the bright bluish tint.

J. H. Chang and co-workers [6] studied direct- and pulse-current (DC and PC) chromium electroplating on rotating cylinder electrode (RCE). The results revealed that current efficiency of hexavalent chromium electrodeposition with DC and PC depends strongly on current density and the rotating speed of the RCE. F. Sanchez Lasheras and co-workers [7] used the design of experiment to improve a neural network model in order to predict the thickness of the chromium layer in a hard chromium plating process. C. A. Huang and co-workers [8] studied the electrochemical behavior of the decorative chromium deposits plated with direct- and pulse-current in 1 M H_2SO_4. The results showed that the surface-crack density of decorative Cr-deposits plated with both DC and PC decreased with increasing the plating current density, whereas the passive current densities in their anodic polarization curves increased when plating current densities increased.

In the present study, Taguchi design [9-17] has been used for understanding the effect of the procedural parameters such as amount of sulfuric and chromic acid, current density and time on thickness of the chromium layers. This method allows for the collection of the essential data for determination of parameters which have the most effect on product quality with a minimum amount of experiment in order to saving time and resources.

1. Experimental

A plating tank of 267 mL equipped with a mixer and air bubbler, heating coil, anodes with the alloy of tin-lead containing 7% tin, digital thermostat, a programmable DC electrical power supply (rectifier), and thickness gauge model DCFN 2000 in order to measuring the thickness of chromium deposit in terms of micrometer were used during the electroplating process.

1.1. Electroplating

The electroplating experiments were carried out using a Watts type bath. Details of the bath composition employed are containing nickel sulfate ($NiSO_4.7H_2O$), nickel chloride ($NiCl_2.6H_2O$) and boric acid (H_3BO_3) with concentrations of 292.5, 40.5 and 31.5 g/L, respectively. All chemicals used in the electroplating experiments were of analytical reagent grade. Solution of sodium hydroxide (0.01N) was used for pH adjustment. The homemade electrochemical cell was employed for nickel electroplating experiments. The dimensions of cell are as follows: 13.9 cm (length) ×12.5 cm (width) ×10 cm (height), corresponding to a volume of 1.75 L. In addition, the cell was equipped with a cathode (made of 99.98% purity copper) and an anode (made of 99.7% purity nickel). The

cathode dimensions are of 8.0 cm (height) ×9.8 cm (length) ×0.1cm (width) while the dimensions of the anode were of 10.0 cm (height) ×4.0 cm (length) ×0.3 cm (width). The immersion surface of cathode was of 0.6dm^2 and respectively the anodic immersion surface was of 0.25dm^2. The above-described cell was connected in galvanostatic regime to the GWINSTEK GPR-1810HD power supply, having a digital control of current and voltage. The temperature of electrolyte solution was kept constant using the Lauda E100 thermostatic bath. Before each electroplating experiment the cathode surface was prepared accordingly. Nickel undercoating is deposited by a nickel electroplating process in one (bright nickel) or two (semi-bright nickel and bright nickel) layers. Nickel provides corrosion resistance and leveling of the substrate surface. Additionally nickel activates the surface and prepares it for chromium plating. The thickness of semi-bright nickel and bright nickel is in a range of 13-30 μm and 5-20 μm, respectively. Decorative chromium is deposited directly on bright underlying nickel. Nickel plated specimens were then double rinsed and subjected to chromium plating process in the experimental setup shown in Figure 1.

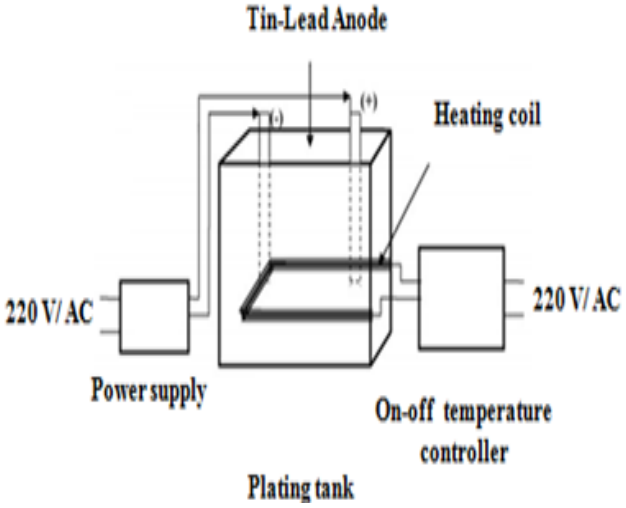

Fig. 1. Schematic diagram of experimental set

The main component of hexavalent chromium plating solutions is chromium trioxide (CrO_3) referred also as chromic acid. The second component is a catalyst, which is either sulfate (SO_4^{2-}) or fluoride. In order to improve the properties of deposits, a set of additives are added to chromium tanks. The composition of these materials is based on organic or inorganic salts (especially salts of alkali or alkaline earth metals which increase the conductivity with a consequent improvement in thickness). In this study, the additives so called **DC** 15 and **DC** 16 (**DC** is an abbreviation of Decorative Chromium built in Schlotter Galvanotechnik of Germany) was added into the tank in order to increasing the thickness of chromium layer. The detailed information about the composition of these materials is shown in Table 1.

Table 1: Composition of applied additives in chromium tank.

Summary Name	Manufacturer Company	Concentration of Chromium
DC 15	Schlotter-Germany	40 g/L
DC 16	Schlotter-Germany	35 g/L

2. Determination of thickness

The control of thickness is of particular importance where the work is to be dyed or where close dimensional tolerances have to be obtained together with adequate protection. Therefore, thickness of deposit may be thought as one of the most important features of the chromium plating process. In this study, the thickness measurement of the specimens was carried out using DCFN 2000 model coating thickness gauge.

3. Results and discussion

3.1. Design of experiment

The quality engineering method proposed by Taguchi is commonly known as the Taguchi method or Taguchi approach [18-20]. His approach provides a new experimental strategy in which a modified and standardized form of design of experiment (DOE) is used. In other words, the Taguchi approach is a form of DOE with special application principles. Taguchi designs experiments using specially constructed tables known as "orthogonal array" (OA). The use of these tables makes the design of experiments very easy and consistent and it requires relatively limited number of experimental trials to study the entire parameter space. As a result, time, cost, and labor saving can be achieved. The experimental results are then transformed into a signal-to-noise (S/N) ratio. Taguchi recommends the use of the S/N ratio to measure the quality characteristics deviating from the desired values. Usually, there are three categories of quality characteristic in the analysis of the S/N ratio, i.e. the-lower-the-better, the- higher-the-better, and the nominal-the-better. The S/N ratio for each level of process parameters is computed based on the S/N analysis. Regardless of the category of the quality characteristic, a greater S/N ratio corresponds to better quality characteristics. Therefore, the optimal level of the process parameters is the level with the greatest S/N ratio. Furthermore, a statistical analysis of variance (ANOVA) is performed to see which process parameters are statistically significant. With the S/N and ANOVA analyses, the optimal combination of the process parameters can be predicted.

According to the Taguchi design methodology, one experimental design should be selected for the controllable factors. The underlying goal of this study is to correlate the thickness of chromium coating to a set of factors such as amount of chromic acid (CrO_3), sulfuric acid, current density and time. The parameters and their levels are listed in Table 2.

Table 3 is an L_{16} orthogonal array (a table of integers whose column elements (1, 2, 3 and 4) represent the levels of each parameter). Each row of the orthogonal array represents a run, that is, a specific set of levels parameters to be tested. The L_{16} orthogonal array (The four-level L_{16} orthogonal array) accommodates four parameters at four levels each in sixteen runs. The orthogonal array used for the optimization of electroplating process in conjunction with their obtained values from thickness measurements in terms of micrometer is displayed in Table 3.

The data obtained from the experiments

Table 2: Suggested Orthogonal design for measurements of thickness and the corresponding experimental thickness (response)

Factors	Level 1	Level 2	Level 3	Level 4
Amount of Chromic Acid (g/l)	150	200	240	280
Amount of Sulfuric Acid (g/l)	0.5	1.0	1.5	1.95
Time (min)	2	4	6	8
Current Density (A/dm^2)	2	4	6	8

may now be analyzed. As it was mentioned in the previous section, Taguchi recommends analyzing the mean response for each run and also suggests analyzing variation using an appropriately chosen signal-to-noise ratio (S/N). For the larger the better responses, the following relation is used for the S/N calculations.

$$SN_L = -10 \log [1/n\ Y_i^2] \quad (1)$$

Where Y_i is the value of the surface thickness for the i-th test.

In our experiment, the system is optimized when the response is as large as possible, so we deal with the SN_L and levels of the parameters that maximizing the SN_L ratios. Table 4 indicates the mean responses of S/N ratios for all of 16 experiments by separated columns for 4 levels of each parameter. It is clear that the second level of time and third level of current density have the most effects on characteristics of the process.

Table 3: L_{16} Orthogonal Array for measurements of thickness.

Run	CrO3	H_2SO_4	Time	CD	Thickness (μm)
1	1	1	1	1	0.39
2	1	2	2	2	0.54
3	1	3	3	3	0.55
4	1	4	4	4	0.30
5	2	1	2	3	0.88
6	2	2	1	4	0.50
7	2	3	4	1	0.36
8	2	4	3	2	0.43
9	3	1	3	4	0.56
10	3	2	4	3	0.52
11	3	3	1	2	0.40
12	3	4	2	1	0.34
13	4	1	4	2	0.61
14	4	2	3	1	0.39
15	4	3	2	4	0.52
16	4	4	1	3	0.55

Table 4: Mean response S/N ratios for thickness power.

Factors	Level 1	Level 2	Level 3	Level 4
Time (min)	-5.588	-2.828	-4.196	-6.287
Amount of Sulfuric Acid (g/l)	-4.510	-5.065	-4.705	-4.619
Current Density (A/dm^2)	-7.437	-4.481	-2.725	-4.255
Amount of Chromic Acid (g/l)	-4.852	-4.855	-4.434	-4.757

The Taguchi method uses graphs of the marginal means of each factor, as shown in Figures 2 and 3. In the Figures, the effects of controllable parameters on mean response and SN_L for thickness measurements are displayed, respectively. In this study, Minitab statistical software was used for the design and analysis of the experiments [21].

According to the previous section, Taguchi-oriented practitioners often use analysis of variance (ANOVA) to determine the parameters that influence the average response and the factors that influence the signal-to-noise ratio [22]. The purpose of the analysis of variance (ANOVA) was to find which parameters significantly affected the quality characteristic. The total sum of square deviation, SS_T can be calculated using (2):

$$SS_T = \sum_i^2 - C.F \quad (2)$$

where, n is the number of experiments in

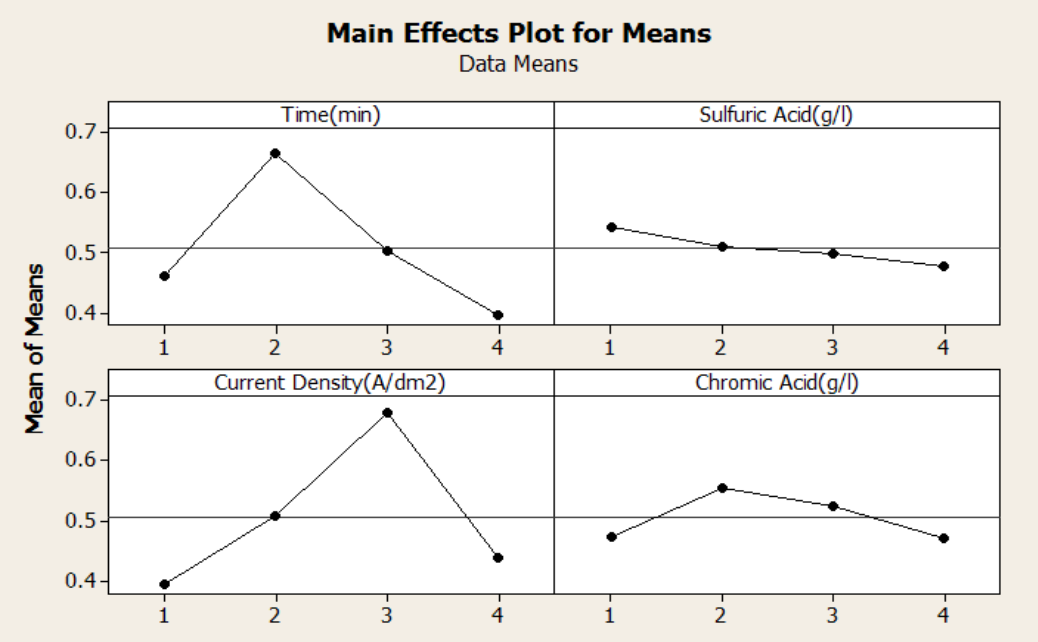

Fig. 2. Main effect plots for mean responses in thickness measurement.

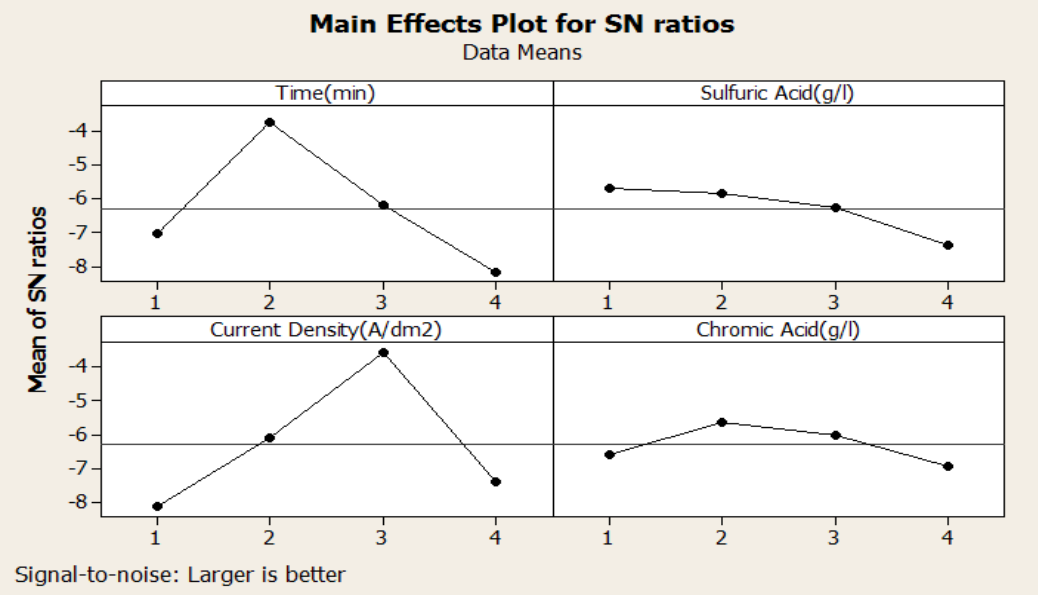

Fig. 3. Main effect plots for signal to noise ratios in thickness measurement

the orthogonal array, Y_i is the total thickness of ith experiment and C.F is the correction factor. C.F was calculated as (3):

$$C.F = T^2/n \quad (3)$$

where, T is the sum of all total thickness. The total sum of square deviations, SS_T was decomposed into two sources: the sum of squared deviation, SS_d due to each process parameter and the sum of square error, SSe. The percentage contribution, P by each of the process parameter in the total sum of square deviation, SS_T was a ratio of the sum of square deviation, SS_d due to each process parameter to the total sum of square deviation, SS_T. Statistically, there is a test called F-ratios (variance ratio) to see which parameters have significant effects on the quality characteristic of thickness. For performing the F test, the mean of square deviation, SS_m due to each process parameter needs to be calculated. The mean of square variations, SS_m is equal to the sum of square deviation, SS_d divided by the number of degree of freedom associated with the process parameters. Then, the F value for each process parameter is simply the ratio of the mean of square deviation, SS_m to the mean of square error, SS_e.

Table 5 represents the ANOVA calculations for thickness measurement. As the Table indicates, the F value of time and current density is greater than the extracted F value of the table for α (risk) = 0.05 (F = 3.29). This means that the variance of these factors is significant compared with the variance of error and the latter factors have a significant effect on the response. In addition, P values of current density and time are smaller than 0.05 which means these parameters are the most influential factors in enhancing the response.

4. Conclusions

This paper has presented an investigation the effect of electroplating parameters on the thickness of chromium layers. The significance of the electroplating parameters on the response was determined using ANOVA. On the basis of ANOVA, the highly effective parameters on thickness were found as time and current density whereas chromic acid and sulfuric acid were less effective factors. Furthermore, the thickness of coating reaches to a high value with a time within 4 minutes and current density of 6 A/dm. Also the Taguchi experimental design approach gives an opportunity to fully understand the effects of process parameters on thickness by performing significantly fewer experiments.

5. Acknowledgment

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Table 5: ANOVA for thickness measurements

Source	DOF	SS	MS	F	P
Time	3	0.1473	0.0491	13.02	0.032
Amount of Chromic Acid	3	0.0095	0.0031	0.85	0.553
Current Density	3	0.1994	0.0664	17.62	0.021
Amount of Sulfuric Acid	3	0.0036	0.0012	0.32	0.813
Error	3	0.0113	0.0037		
Total	15	0.3712			

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