

Analysis of the Characteristics of Nickel-Plating Baths

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Abstract

Nickel plating processes, popularly used in Europe since 1950s, have been increasingly employed in Turkey in recent years. As a result, industrial usage has developed rapidly. Nickel plating is the preferred process for this study because of the uniformity of the plating thickness on the plated surface and the ease with which complex components can be plated. It is also resistant to corrosion and has good levels of hardness.

In this experiment, the following parameters were investigated: the effect of varying the amount of nickel, the thickness of the plating, and sheen (whether shiny or dull).

In this study, brightening and carrying agents have been used to determine the best operational parameters for the Kale Kilit Factory. The compositions of the brightening and carrying agents used in the experiment are included in this text. Additionally, investigations were conducted on how the plating color changed when compared to the original bath as a result of using Fe^{2+} complex concentration in nickel plating baths, as a brightening agent and for analyzing the color of the plated sample, under a visible region spectrophotometer at 350 nm and 6500 kelvin light power and D65 (Average North Sky Daylight) light. In our research, the most important feature of analysis is that the desired color effect is obtained by using brightening agents containing Fe^{2+} complex concentration without any interference from any colorant after nickel plating by electrolysis. In this study where nickel plating bath characteristics are researched, physical tests such as brittle test are applied to the plated materials, which provide us important ideas about the accuracy of the choices made according to standards.

Keywords: Nickel plating, Electrolytic plating, Corrosion, Electron microscopy, X-ray unit, Visible region spectrophotometer, Brightening agent, Carrying agent

1. Introduction

Various types of plating processes are extensively employed in different industries, both to increase the resistance to corrosion and to impart a more decorative appearance to steel and to metals such as aluminum, zamak, and brass. Second, and the most frequently used plating process is nickel plating (with zinc plating being the most popular). As nickel alone does not provide the desired visual effects, it is plated over with chromium in most cases.

When applied to hard and soft metals, nickel plating does produce a distinct decorative appearance and provide corrosion resistance; however, best results have been obtained with chromium plating over sulfate- based nickel plating.

2. Experiments

In this experiment, the following parameters of the nickel plating process were investigated: amount of nickel, thickness of the plating, and sheen (whether shiny or dull).

2.1 Experimental Conditions

In the present study, the “Kale Kilit and Kalıp Sanayi A.Ş.” Chemistry Laboratories and their plating facilities were selected as the environment for the experiments. In addition to nickel baths, the Kale Kilit A.Ş. plating facility possesses cyanide copper baths, chromic acid baths, zinc acid baths, brass sulfate baths, and brass cyanide baths, all of 1- ton capacity.

2.2 Choice of the Chemicals

Watt- type baths (Saticioglu, 2006, pp. 22-25; Yilmaz, 2006, pp. 36-44) are used in all nickel processes, indicating that the inorganic compositions resemble one another. Therefore, the main composition of the nickel-plating bath in our experiments was as in Table 1, and the analytical composition of the bath was as in Table 2.

2.3 Hull Cell

In 1937, the American scientist A. Hull analyzed the link between anode-cathode spacing and the current density on a piece of metal and developed a formula based on his findings. Hull then designed a special container with this information in mind. Today, both the experiment and the container are referred to as Hull Cells (Cetinkaya, 2006, pp.34-36). The Hull Cell container is a miniature plating unit consisting of cathodic plating on a metal plate, designed to test the bath and the plating characteristics. The interpretation of the plated test panel provides data on the brightness, homogeneity, plating quality, distribution power, contamination, and the chemistry of the plating bath. The Hull Cell reflects the actual process that occurs in the plating bath in the context of the proposed operational parameters. The plated specimen and the Hull Ruler are together used to carry out the research. Thus, preventative maintenance, error correction, and quality control functions are tested and rapidly performed without damaging the actual bath. All plating in this study was done using a Hull Cell.

2.4 Preliminary Preparation for the Experiment

The plating experiments were performed using Hull Cell (HC) containers. The HC work was done at 50°C, without mixing. In each case, shiny stainless steel plates of 70 cm² (10 cm * 7 cm) surface area were used. A current of 1 A, (0.01 A/cm²), was applied to the plates for 10 minutes. However, since the plating was not sufficiently thick, a bath with the same chemical composition was used for a second time, with the current increased to 2 A, (0.02 A/cm²), for 10 minutes. The plates were cleaned in an degreasing electrolytic bath prior to plating and were rinsed with tap water. They were then neutralized in a 10% sulfuric acid bath prior to being rinsed once again with tap water. The plates were then subjected to the Hull Cell operation without being dried.

2.5 Hull Cell Test

Initial experiments were performed without adding organic chemicals (such as a carrying or a brightening agent) to the electrolyte solution (Figure 1).

2.5.1 The Effect of the Carrying Agent

The composition of the carrying agent is shown in Table 3.

We investigated varying concentrations of the carrying agent: 5 g/L (Figure 2), 10 g/L (Figure 3), and 15 g/L (Figure 4). Anticipating the quantities that would be required in later experiments, as well as, other influences, the 10 g/L amount was chosen and then further increased by 10% to 11 g/L. An 11 g/L concentration of carrying was added to the solution and the test panel was plated by the Hull Cell. The resultant plating had a matte texture. Following X-ray examination, it was determined that plating ranging between 1 and 5 microns in thickness was achieved over the investigated current range. As good results were obtained with the 11 g/L carrying agent concentration, this was selected as the most suitable concentration from a cost perspective.

2.5.2 The Effect of the Brightening Agent

The composition of the brightening agent is shown in Table 4.

The concentrations of the brightening agent used were 0.3 g/L (Figure 5), 0.5 g/L (Figure 6), and 0.7 g/L (Figure 7). From this survey, for the desired color difference, the optimal concentration of brightening agent to be used was defined as 0.5 g/L according to the fragility test results. The repeated addition of 6 g/L Ni²⁺ to the bath resulted in reduced brightness. Accordingly, it was decided to maintain the Ni²⁺ density at 75 g/L. A high percentage of nickel in the plating caused brittleness of the plating. The quantities of the brightening agent and other compounds were determined in separate instances. To ensure a standard concentration of the various compounds used a large volume of the mixture. Thus, the proportions were kept constant, irrespective of the quantity that was added to the bath.

2.6 Results of the X-Ray Examination

The X-ray equipment (Fischer Instruments GmbH, 2002) measures the plating thickness and provides accurate information on the Ni percentages, as well as the average, maximum and minimum values, and the standard deviation.

The plating thickness of the nickel-plated iron, plated in the bath, was measured by a Helmut Fischer brand X-ray coating thickness measurement gauge. The results are given in Table 5, Table 6, Table 7, Table 8, Table 9, Table 10, Table 11, Table 12, Table 13.

The nickel plating thickness standard for the lock components at the Kale Kilit Factory was set in range of 10-20 microns. The nine plated pieces complied with these standards.

2.7 Nickel-Plating Bath Analyses

Necessary Materials

25% Ammonia solution ($\text{NH}_3/\text{H}_2\text{O}$, pH=11.7), 10% Potassium chromate solution, Murexide indicator, Boric acid buffer solution, 0.1 M EDTA, 0.1 N Silver nitrate, 0.2 N Sodium hydroxide.

2.7.1 Metallic Nickel Analysis

Using a pipette, 1 g of sample was transferred from the bath into an Erlenmeyer flask. Next, 100 g of pure water, 10 g of ammonia (25%, pH=11.7), and a small quantity of murexide indicator were added. This was titrated with EDTA until a color change was observed.

Result: Consumption $\times 5.87 = \dots$ g/L metallic nickel (Ni^{2+})

2.7.2 Chloride Analysis

A 1 g sample was pipetted from the bath and transferred into an Erlenmeyer flask. Next, 100 g of distilled water and four or five drops of potassium chromate were added, and the color changed to crimson-red. It was then titrated with silver nitrate until it became reddish-brown in color.

Result: Consumption $\times 3.75 = \dots$ g/L chloride (Cl^-)

2.7.3 Analysis of Boric Acid

A 1 g sample was pipetted from the bath and transferred into an Erlenmeyer flask. Next, 25 g of boric acid buffer solution was added. This was titrated with sodium hydroxide until a violet color was achieved.

Result: Consumption $\times 6.2 = \dots$ g/L boric acid (H_3BO_3)

The experimental results are given in Table 14.

3. Discussion

Recently, the decorative use of nickel plating baths in the plate industry has caused the industry itself to seek new waves of research. One of those states that instead of applying colorants according to the desired colors after the metal coating for corrosion protection and decorative purposes, simply add brightening agents, simultaneously to the metal plating bath solutions, as colorants. In our studies, the results we obtain from different proportions, according to the desired colorant effect, is measured with electron microscopy, hardness durability, X-ray method (to measure the thickness of plate), and visible spectrophotometry.

When analyzing with the electron microscope, Jeol 6060 Scanning Electron Microscope is used. The working range of this microscope is; high voltage separation power 1~ 30 kV, magnification 10~ 1 000 000, using SEI mode (secondary electron image)~ BEI (backscattered electron image), its resolution is 30 kV (SEI) at 1.2 nm, and 30 kV (BEI) at 3.0 nm. Also the listed parameters of the electron microscope with regard to image definition and working quality are determined as follows: resolution 100 nm, high voltage separation 8.0 kV, using SEI mode, working distance (WD) 8.1 mm, and a magnifying scale of 80 000. To produce images of surfaces, SEI is used as a first choice because in electron microscopes, the electrons sent on to the specimen produce two kinds of images. The first is the secondary electron image (SEI), which is used to produce images of secondary electrons that originate after stimulating specimen atoms, and the second is the backscattered electron image (BEI), which consists of the electrons that are backscattered out of the specimen interaction volume by elastic scattering interactions with specimen atoms. SEI uses a topographical contrast, whereas, BEI creates the contrast according to the atomic numbers. In other words, with SEI it is possible to examine the sample surface with regard to its form and with BEI with regard to the chemical differences. This is why we have used secondary electron images (SEI) in our studies.

In total we have carried out our research with seven bath samples, which consists of one original, three different proportions of nickel baths including carrying agent chemicals, and three different proportions of nickel baths with brightening agent chemicals, thus acquiring seven electron microscope images. As can be seen from the electron microscope images, the concentration of carrying agent chemicals does not have a significant effect on the surface, whereas, the concentration involving brightening agents, with the proportions rising, the color of the nickel plated metal surface transforms into the different tones of red, because the brightening agent has metallic iron (from figure 1 to figure 7).

The impact of the Fe^{2+} complex present in the brightening agent on the plating surface is measured with CIE L^*a^*b color space (Yilmaz, 2002, pp.340-350; Dogan, & Zaimoglu, 2006, pp.107-118). CIE L^*a^*b color system is the most used space. Measurement of the comparison of color values is more recognizable with this system. 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 (Icoglu, 2006, pp.41-43), L^* , a^* , b^* , c^* , and h^0 values are the main parameters used in defining the colors.

Where

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

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

c^* : Is named as chromaticity. The chromaticity value of the color increases as the value drifts from the center of the color space in a horizontal level. It gives information about the saturation of the color. A color and its tone at the same level is the characteristic defining its distinction level from another color (between black and white). If a color diverges from black, its saturation will increase, if it approaches to gray, its saturation will decrease. Exact gray's saturation is 0. As 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): Is the basic color of an object (like red, blue, yellow), it is an angle between c^* (chromaticity) and the axis of abscissa. Angle h^0 is 0° and 360° along $+a^*$, 90° for $+b^*$, 180° for $-a^*$, and 270° for $-b^*$. It gives an idea about the size of the color. The h^0 value can be defined as the angle between the axis of the abscissa of measured color and axis of the abscissa of standard color (Figure 8).

The colorant effect of the chemical, which contains the Fe^{2+} complex added to the nickel plating bath, on the metal plate is examined with the Macbeth CMC-CE-7000-XL0339 which is a visible region spectrophotometer. The device is at 325 nm wavelength and capable of taking measurements in the range of 2000~7500 kelvin light power. D65 daylight used in the spectrophotometer is equal to 6500 kelvin light power, and this light power means light power for the plated material surfaces. These values are decided according to European and Japanese standards. According to the information gathered from the result of this analysis, under D65 daylight measurement, it is observed that brightening agent plates located on the red side of the red~green axis of the illuminant D65 graphics result in an increase in the red axis when the brightening agent concentration is increased. Similarly, during addition of carrying agent, it is observed that the sample plate in illuminant D65 graphic does not diverge from the standard (original) bath plate axis (from figure 9 to figure 14). It is observed that the h^0 (hue) and c^* (chromaticity) values of the samples do not change much at the point of saturation and hue value of red color. Similarly, the L value, which can be expressed as lightness~darkness, is observed and does not change with addition of brightening agent and carrying agent, and is shown as a 'light~dark' scale at each graphic axis. At the measurement of the L value, the distance to the standard rectangle located at the middle of the light~dark scale is referenced.

When deciding the optimum working conditions for nickel baths which are the main topic of our study, effects of the medium with added chemicals on the bath's physical features are researched. As for this, effects of nickel baths with increasing pH, due to added brightening agent, on the working quality of the baths are researched by brittling tests of the plated materials. Increase of the brightening agent amount added to the bath raises the pH of the bath, as is shown in the X-ray report. Increase of pH in the bath causes more fragility in the plating. The 'Increasing Progressive Power Test Method' (Kiray, 2007, pp.313) is used in brittle tests as the measurement method. In this method, power is applied to a plate that is progressively between two plates. For iron platings, 14.701 N power is applied, which is acceptable by ASTM 8 standards. A bath plating material with 5.5 pH cracked after 15 hours; a plate with 6 pH cracked after 14.9 hours; and a plate with 6.5 pH cracked after 10 hours of the test.

Visible region spectroscopy:

For visible region spectroscopy, two different measurements are taken from each plate surfaces. Therefore, these measurements are figured as 2 in the light axis. The original axis ($-a, +a$ and $-b, +b$) whose every aperture is figured as 1, means the color measurement of the plate is taken from the visible the region spectroscopy and does not contain any brightening agent or carrying agent. Other axes in the arch form shows color measurements of plated materials with nickel bath to which brightening and carrying agent are added at different proportions for determining h^0 and c^* parameters. Therefore, the parameters for appointing color for standard (original) bath are calculated as follows (Dogan, & Zaimoglu, 2006, pp.107-118);

$$\Delta L^* = L^*_{\text{sample}} - L^*_{\text{standard}}$$

$$\Delta c^* = c^*_{\text{sample}} - c^*_{\text{standard}}$$

$$\Delta h^0 = h^0_{\text{sample}} - h^0_{\text{standard}}$$

Color measurements and the results of calculations of the color measurements are given in Table 15, Table 16, Table 17, Table 18, Table 19, Table 20, Table 21, Table 22. All measurements given in tables are color measurements which were gathered with the original bath taken as the standard.

4. Conclusion

The effects of the carrying and brightening agents were observed under laboratory conditions. In this study, it was observed that the carrying agent facilitated the deposition of nickel onto the surface and increased cohesion, thus resulting in coating of all surfaces of the component to be plated. This has been confirmed by thickness reports from X-ray equipment and observations by electron microscopy. The brightening agent reduced the productivity of the current. All of these results could have been obtained from thickness reports of the X-ray unit because, according to these reports, plating thickness was decreased. When carrying and brightening agents were added, it was observed the brightening agent increased the pH level of the nickel bath, but the carrying agent did not affect the pH levels. In the bending experiments that were performed on a coated plate, after adding a brightening agent, increased brittleness was observed. On the other hand, the desired decorative appearance was obtained. Current density was as 0.02 A/cm² because plating thickness of 10-20 microns, the determined quality standard at the Kale Kilit factory has been obtained at this current density. The conducted experimental work determined the optimum quantities of both organic compounds. It was concluded that the optimum quantity of the carrying agent was 11 g/L, and of the brightening agent, 0.5 g/L.

After the study, the main composition of the nickel bath was determined to be as follows:

260 g/L Nickel sulfate (NiSO₄ .6H₂O)

67 g/L Nickel chloride (NiCl₂)

45 g/L Boric acid (H₃BO₃)

Chemicals:

11 g/L Carrying agent

0.5 g/L Brightening agent

The optimal working parameters for the factory are as follows:

45°C–55°C Temperature

0.01-0.05 A/cm² Current Density

0.44 micron/min. (at 0.02 A/cm²) plating speed

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Table 1. The main composition of the nickel-plating bath

Material Name	Formulation	Concentration (g/L)
Nickel sulfate	$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	260
Nickel chloride	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	67
Boric acid	H_3BO_3	45

Table 2. The analytical composition of the bath

Material Name	Formulation	Concentration (g/L)
Metallic nickel	Ni^{2+}	75
Chloride	Cl^-	20
Boric acid	H_3BO_3	45

Table 3. The composition of the carrying agent

Material Name	Formulation	For a 100 kg product
Sodium saccharine	$\text{C}_7\text{H}_4\text{NNaO}_3\text{S} \cdot 2\text{H}_2\text{O}$	25.0 kg
35% Sodium allyl sulfonate	$\text{C}_3\text{H}_5\text{NaO}_3\text{S}$	6.7 kg
Water	H_2O	68.3 kg

Table 4. The composition of the brightening agent

Material Name	Formulation	For a 100 kg product
Metasulfobenzaldehyde	$\text{HSO}_3\text{C}_6\text{H}_5\text{CHO}$	7.6 kg
Pyridine-3-sulfonic acid	$\text{SO}_2\text{OH}_3\text{C}_5\text{H}_5\text{N}$	3.8 kg
p-vinylbenzenesulfonic acid	$\text{C}_6\text{H}_5\text{CHCH}_2\text{C}_6\text{H}_4\text{O}_3\text{S}$	1.9 kg
o-benzoylsulfonamide	$\text{C}_6\text{H}_5\text{SO}_2\text{NH}_2$	8.6 kg
Sodium saccharine	$\text{C}_7\text{H}_4\text{NNaO}_3\text{S} \cdot 2\text{H}_2\text{O}$	10.0 kg
Iron (II) acetylacetonate	$\text{CH}_3\text{COCH}=\text{C}(\text{O})\text{CH}_3)_2\text{Fe}$	0.3 kg
Water	H_2O	67.8 kg

Table 5. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.1

Application: 1/Ni/Fe -0.01 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	6.1	7.2	8.5	7.26	1.513	20.84	2.4	8.5	6.1	10

Table 6. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.2

Application: 1/Ni/Fe -0.02 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	10.2	11.4	13.7	11.76	2.206	18.758	3.5	13.7	10.2	10

Table 7. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.3

Application: 1/Ni/Fe -0.03 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	18.7	21.4	25.6	21.9	4.350	19.86	6.9	25.6	18.7	10

Table 8. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.4

Application: 1/Ni/Fe -5 g/L carrying agent, 0.02 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	11.3	15.1	16.8	14.42	2.837	19.68	5.54	16.8	11.3	10

Table 9. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.5

Application: 1/Ni/Fe -10 g/L carrying agent, 0.02 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	11.7	13.2	16.2	13.7	2.453	7.6	2.54	16.2	11.7	10

Table 10. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.6

Application: 1/Ni/Fe -15 g/L carrying agent, 0.02 A/cm^2 current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	15.4	18.9	18.6	17.62	1.939	11.01	3.52	18.6	15.4	10

Table 11. Product: 1/Ni/Fe Dir.: Fischer Block: 3.7.7

Application: 1/Ni/Fe – 0.3 g/L brightening agent, 0.02 A/cm² current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	16.7	18.2	20.1	18.33	1.85	15.47	3.4	20.1	16.7	10

pH: 5.5

Table 12. Product: 1/Ni/Fe Dir. Fischer Block: 3.7.8

Application: 1/Ni/Fe – 0.5 g/L brightening agent, 0.02 A/cm² current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	11.4	15.7	16.3	14.42	3.08	21.42	4.9	16.3	11.4	10

pH: 6

Table 13. Product: 1/Ni/Fe Dir. Fischer Block: 3.7.9

Application: 1/Ni/Fe – 0.7 g/L brightening agent, 0.02 A/cm² current density

Number of Reading	Nickel Plating Thickness (μm)			Mean (μm)	Standart Deviation (μm)	C.O.V (%)	Range (μm)	Maximum Reading (μm)	Minimum Reding (μm)	Measuring Time (Sec.)
3	8.3	9.5	10.1	9.3	1.13	12.20	1.8	10.1	8.3	10

pH: 6.5

Table 14. The experimental results

Material	Consumption		Material	Consumption		Material	Consumption
Ni ²⁺ (g/L)	EDTA (g)		Cl ⁻ (g/L)	AgNO ₃ (g)		H ₃ BO ₃ (g/L)	NaOH (g)
70.44	12		24.85	6.6		37.2	6

Table 15. Color measurement of plated sample (material) with bath which does not contain brightening and carrying agent

Standart	Illuminant	L*	c*	h ⁰
Original bath	D65	19.664	22.631	17.213

Table 16. Color measurement of 5 g/L carrying agent added plating bath (Figure 9)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	19.659	22.609	17.200
Trial 2	D65	19.645	22.599	17.188

Table 17. Color measurement of 10 g/L carrying agent added plating bath (Figure 10)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	19.643	22.625	17.205
Trial 2	D65	19.626	22.618	17.206

Table 18. Color measurement of 15 g/L carrying agent added plating bath (Figure 11)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	19.622	22.617	17.198
Trial 2	D65	19.624	22.611	17.166

Table 19. Color measurement of 0.3 g/L brightening agent added plating bath (Figure 12)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	18.342	23.530	19.366
Trial 2	D65	18.205	23.444	19.506

Table 20. Color measurement of 0.5 g/L brightening agent added plating bath (Figure 13)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	18.135	23.410	19.436
Trial 2	D65	18.027	23.261	19.618

Table 21. Color measurement of 0.7 g/L brightening agent added plating bath (Figure 14)

Sample (Material)	Illuminant	L*	c*	h ⁰
Trial 1	D65	18.299	23.368	19.320
Trial 2	D65	18.283	23.298	19.584

Table 22. Results of calculations of the color measurements

Sample (Material)	Trial 1			Trial 2		
	ΔL^*	Δc^*	Δh^0	ΔL^*	Δc^*	Δh^0
5 g/L carrying agent added bath	-0.005	-0.022	-0.013	-0.019	-0.0232	0.025
10 g/L carrying agent added bath	-0.021	-0.006	-0.008	-0.038	-0.013	-0.007
15 g/L carrying agent added bath	-0.042	-0.014	-0.015	-0.04	0.02	-0.047
0.3 g/L brightening agent added bath	-1.322	0.899	2.153	-1.459	0.813	2.293
0.5 g/L brightening agent added bath	-1.529	0.779	2.223	-1.637	0.630	2.405
0.7 g/L brightening agent added bath	-1.365	0.737	2.107	-1.381	0.667	2.371

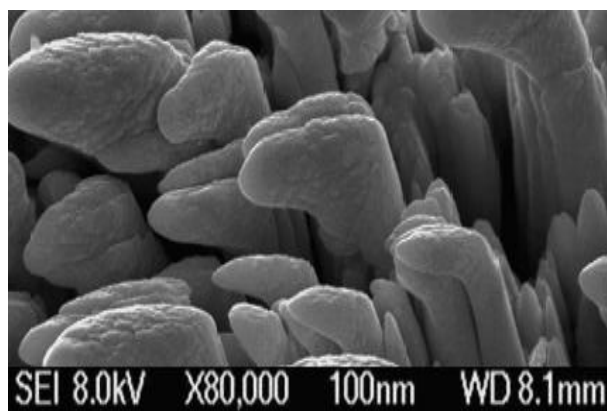


Figure 1. The SEM image of the original form of the nickel plating bath

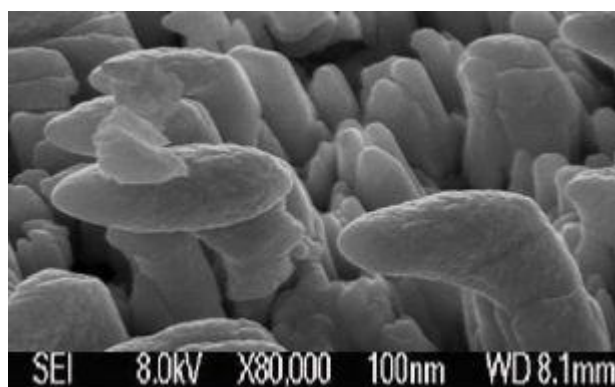


Figure 2. The SEM image of 5 g/L carrying agent added plating bath

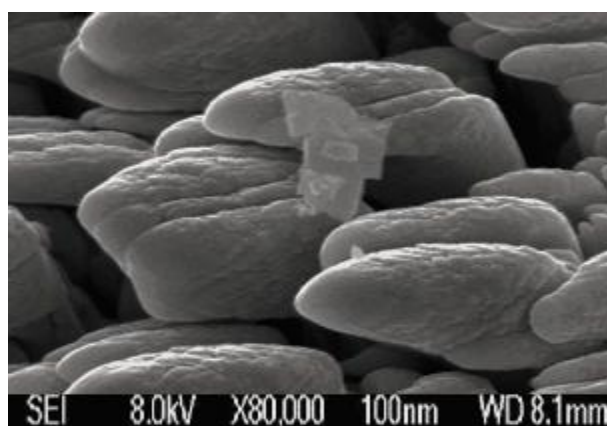


Figure 3. The SEM image of 10 g/L carrying agent added plating bath

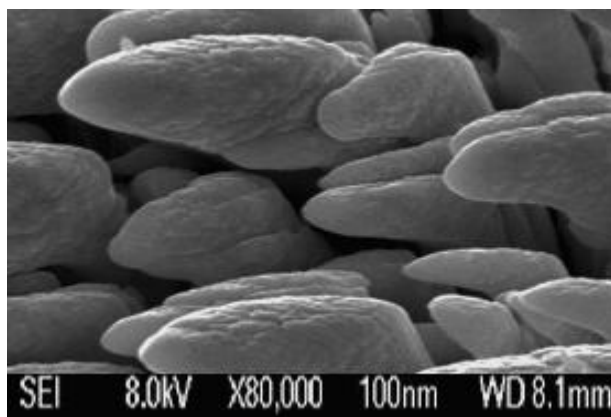


Figure 4. The SEM image of 15 g/L carrying agent added plating bath

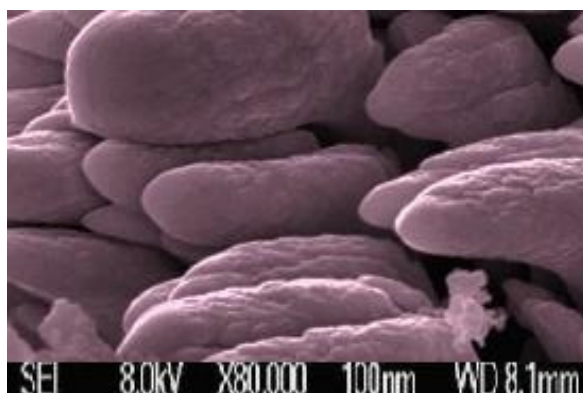


Figure 5. The SEM image of 0.3 g/L brightening agent added plating bath

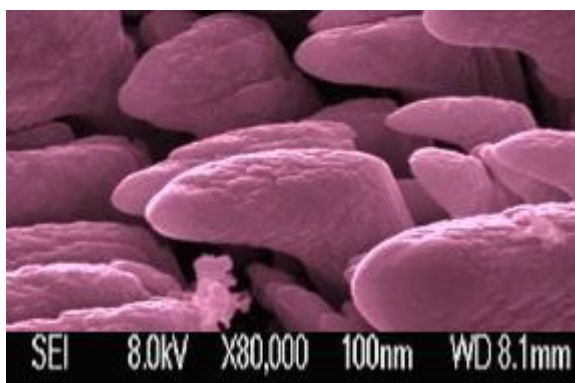


Figure 6. The SEM image of 0.5 g/L brightening agent added plating bath

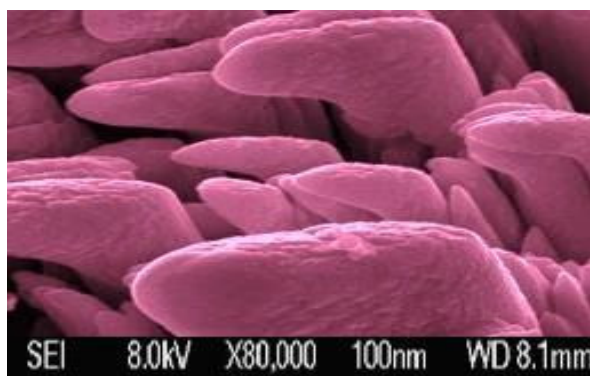


Figure 7. The SEM image of 0.7 g/L brightening agent added plating bath

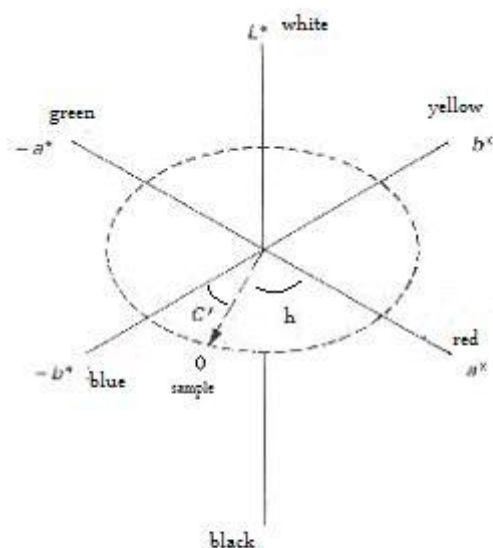


Figure 8. CIE L*a*b* Color Space

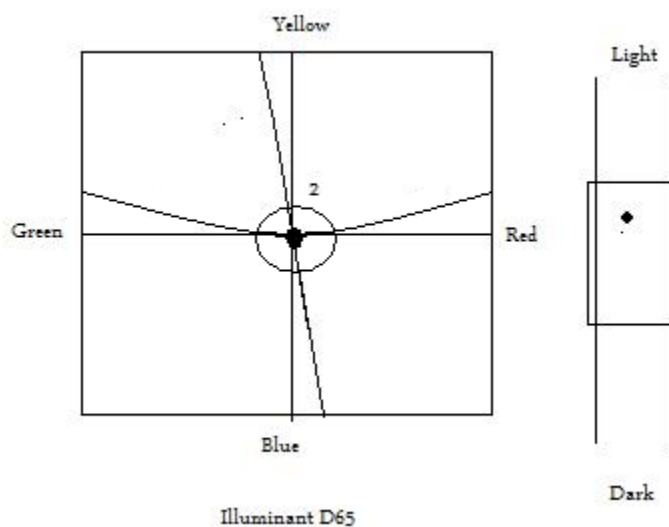


Figure 9. Color measurement of 5 g/L carrying agent added plating bath

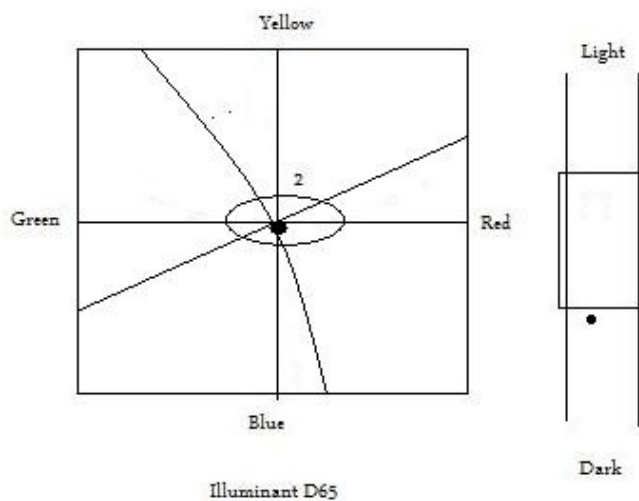


Figure 10. Color measurement of 10 g/L carrying agent added plating bath

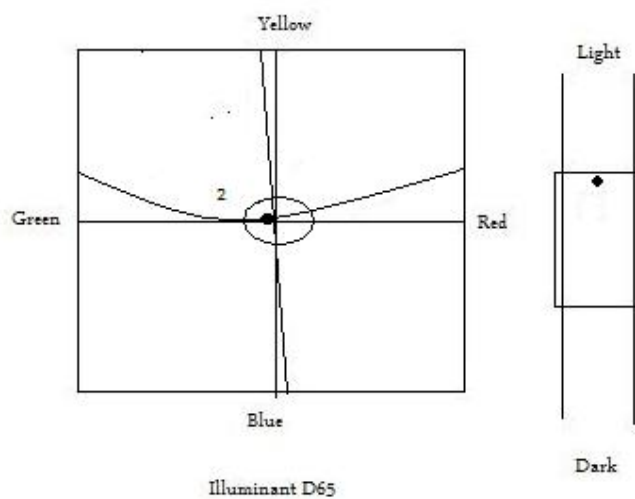


Figure 11. Color measurement of 15 g/L carrying agent added plating bath

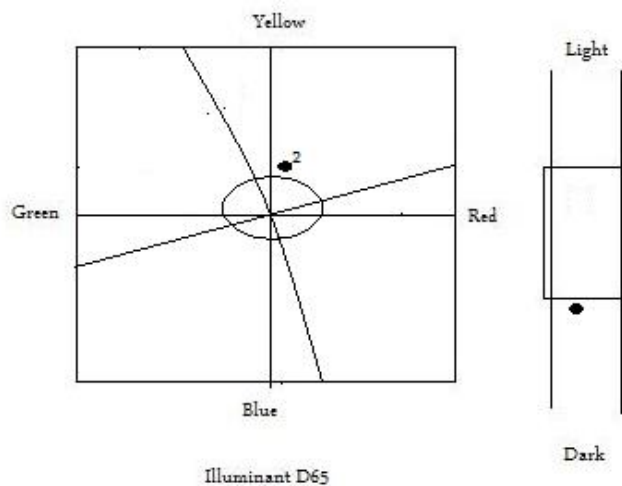


Figure 12. Color measurement of 0.3 g/L brightening agent added plating bath

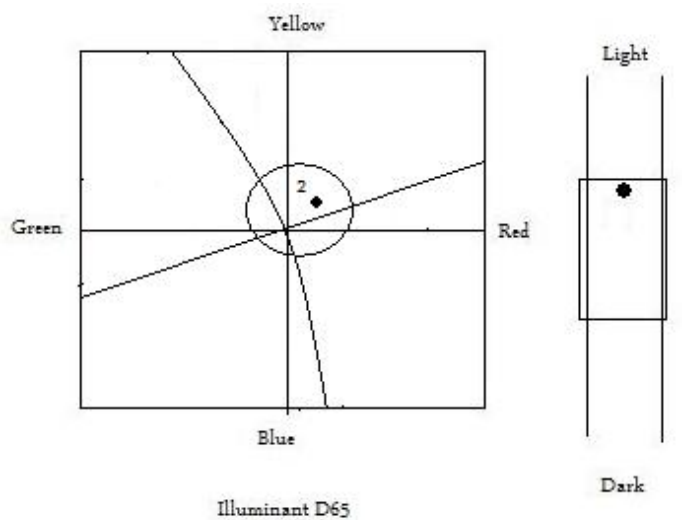


Figure 13. Color measurement of 0.5 g/L brightening agent added plating bath

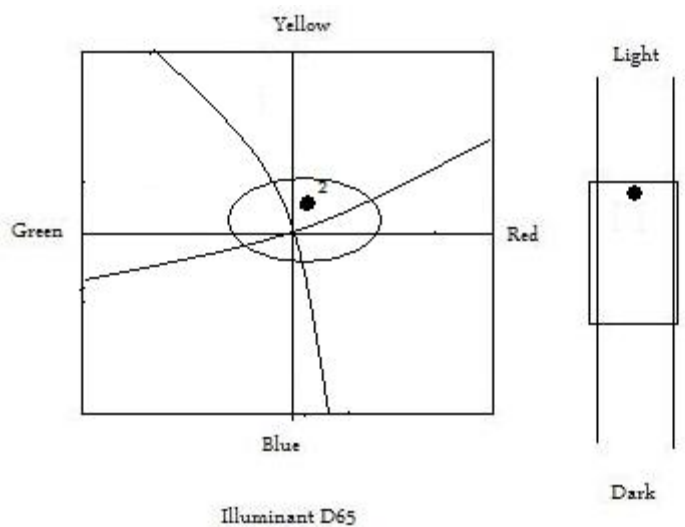


Figure 14. Color measurement of 0.7 g/L brightening agent added plating bath