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The effect of saccharin addition to nickel electroplating on the formation of nanocrystalline nickel deposits

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Abstract. This study investigates the effect of saccharin addition on nickel deposits. Coating substrates with nickel is carried out through electroplating. This method involves Watts bath, 0, 1, 3, and 5 g/L saccharin, and a current density of 6 A/dm^2. The electroplating process lasts for 20 minutes at 50ºC. The plated substrate is a Steel Plate Cold Rolled Coil (SPCC). The deposits attached to the SPCC surface is characterized by a Scanning Electron Microscope (SEM) to observe the structural changes of the deposits and an X-Ray Diffraction (XRD) to calculate their grain size. The results show that, first, nickel deposits without saccharin addition has a pyramidal and crystal polyhedral structure while those with saccharin reflect a globular structure and colonial patterns. Secondly, the addition of 5g/L saccharin produces the smallest grain, 17.39 nm.

1. Introduction

Coatings are often and easily applied to prevent metal corrosion. This technique has several variants such as electroplating, Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD), laser beam deposition, and ion implantation. Of all the types of coating, electroplating is the most economical and its production rate is so high that this method fit the use in industry [1]. On the other hand, the material for coating or plating the object is nickel and thus, the method is completely named 'nickel electroplating'.

Nickel electroplating is the process of depositing nickel onto the surface of a conductive material electrochemically through direct electric current. This technique gets the metal surface more shining and more resistant to corrosion in the atmosphere [2]. Nanocrystalline nickel deposits can be produced through electroplating which results in low porosity residuals [1]. In addition to enhancing corrosion resistance, the nano-sized nickel grains also raise the metal strength and tribological properties [5].

The scale of nanometer on the deposits can be obtained by adding saccharin to nickel electroplating. Besides electroplating, several parameters capable of producing nano-sized particles include electrodeposition mode, temperature, current density, and stirring [5, 6, 7]. Here are some previous investigations that involve saccharin and the aforementioned parameters. Rashidi and Amadeh (2008) found that the addition of 5 g/L saccharin in Watts bath reduced the nickel grain size, from 182 nm to 24 nm, by increasing the current density from 10 to 75 mA/cm^2 [1]. Next, El-Sherik (1995) concluded that the decline in nickel electrodeposition grain size between 40-10 nm occurs when saccharin from 0.5 to 10 g/L is put to Watts bath, using pulsed current [5].

This research explores the effect of saccharin addition to nickel electroplating on nickel deposits. The characterization of nickel particles uses a Scanning Electron Microscope (SEM) to observe their structural changes and an X-Ray Diffraction (XRD) to calculate their grain size. The object to plate is a Steel Roll Cold Rolled Coil (SPCC).

2. Methodology

2.1. Materials

The plated material in this study is a 50x50x2 mm Steel Plate Cold Rolled Coil (SPCC) which plays as a cathode. SPCC is a carbon steel produced through cold rolling and extensively exploited in automotive and electrical equipment. Carbon rods perform the function as an anode. Figure 1 below is the picture of a steel plate cold rolled coil surface.

Figure 1. SPCC Substrate.

Table 1 lists the results of SPCC substrates composition analysis through an Optical Emission Microscope (OES). The results have satisfied the JIS G3141 standard: commercial quality, where the contents of carbon are less than 0.12%.

Table 1. SPCC Chemical Composition.

Material	Composition			
	C (max)	Mn (max)	P (max)	S (Max)
SPCC	0.0482	0.182	0.011	0.0079

2.2. Research Procedure

Surface preparation is the initial stage of nickel electroplating. This preparation aims to remove rust, oil, and other inhibiting substances from the surface of the substrate. The stages in the steel surface preparation are as follows. First, the surface was rubbed with a sandpaper, grit of 120-1000, to get rid of the oxide layer and dirt. Then, the object underwent the 10% NaOH-degreasing process for 11 minutes at 65ºC. Next, the surface was 32% HCl-pickled for 10 minutes at a room temperature. Lastly, the substrate surface was etched with 10% H_2SO_4 for 4 minutes at a room temperature.

After the surface preparation, the process came to nickel electroplating. This process engaged (**a**) Watts bath solution containing 300 g/L nickel sulphate ($NiSO_4$), 30 g/L nickel chloride ($NiCl_2.6H_2O$), and 30 g/L boric acid (H_3BO_3), (**b**) 0.2 cc/L wetting agent, as well as (**c**) 0, 1, 3, and 5g/L saccharin. The pH of the solution ranges from 4 - 4.2. This plating process lasted for 20 minutes at 50ºC with a current density of 6A/dm^2. The process then left nickel deposits on the substrate surface. Nickel deposits were then observed with an optical microscope, a Scanning Electron Microscope (SEM), and an X-Ray Diffraction (XRD) to find the form and pattern of the nickel deposits and their grain size.

Only in XRD, the observation collaborates with Schererr equation. The formula for this equation is provided bellow:

$$D = \frac{k\gamma}{\beta \sin\theta} \quad (1)$$

D is the grain size at the nanometer scale, *k* the Scherrer constant, *γ* the X-Ray wavelength, and *β* the Full Width at Half of Peaks (FWHP). The analysis of grain size through Scherrer equation was conducted with the help of an X'Pert High Score Plus.

3. Results and Discussion

This chapter is comprised of such sub-chapters as optical microscopic observation, Scanning Electron Microscope (SEM) observation, and X-Ray Diffraction (XRD) observation.

3.1. Optical Microscopic Observation

Figure 2a shows the results of optical microscopic observation on the macro photography of nickel-electroplated steel without saccharin addition. On the contrary, those with saccharin addition is on figure 2b (1 g/L), c (3 g/L), and d (5 g/L).

The no-saccharin addition nickel surface has a darker surface than the saccharin-added nickel layer, as shown on figure b, c, and d. After being investigated, the saccharin-added nickel layers are smoother than that which is not saccharin-added. Inside the finer nickel layers exist very small grains which have the light bounce at the same angles. Therefore, the surface appears brighter. Meanwhile, the rough surfaces get the light to scatter to different angles and thus, look more gloomy [8]. In other words, the addition of saccharin to nickle electroplating leads to smaller and smoother nickel particles and even layers.

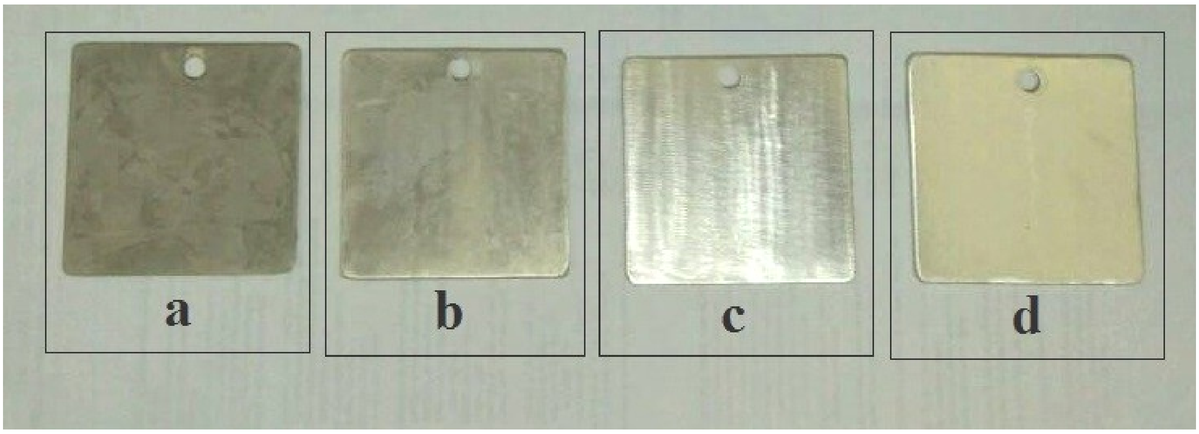

Figure 2. The optical microscopic observation on the macro photography of nickel-electroplated steel with saccharin addition; a) 0 g/L, b) 1 g/L, c) 3 g/L, and d) 5 g/L.

Figure 3 presents the results of optical microscopic observation on the 'cross-section' macro photography. The formed nickel layers were examined using an optical microscope at 200x magnification. Figure 3a, b, c and d show that the substrates have been completely plated with nickel either with the addition of saccharin or not. Nickel coatings look yellowish-white while SPCC is shown in ferrite (bright) and perlite (dark)-phased macro structures.

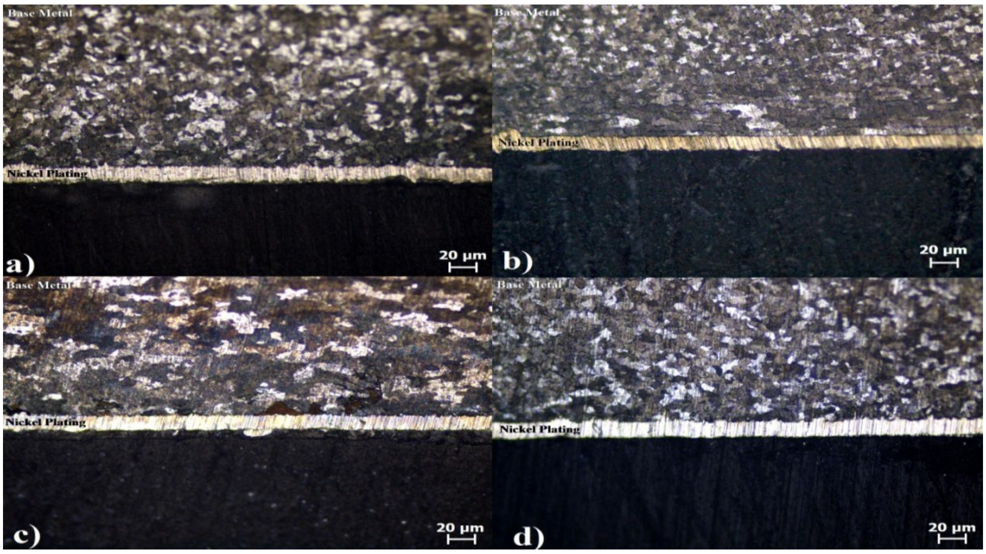

Figure 3. The cross-section of nickel-electroplated steel with saccharin addition; a) 0 g/L, b) 1 g/L, c) 3 g/L, and d) 5 g/L at 200x magnification.

3.2. Scanning Electron Microscope (SEM) Observation

The observation of the form of nickel deposits uses a scanning electron microscope at 5000x magnification. The results are shown on figure 4. On the one hand, figure 4(a) presents that no-saccharin addition nickel coatings have pyramidal and polyhedral crystals and an uneven surface.

On the other hand, the addition of saccharin to nickel electroplating as shown on figure 4(b), (c), and (d) produces globular and colonial particles. The formation of colonies on these deposits improves the density between particles. This phenomenon is an inhibitory effect of organic additives on the reduction of deposited nickel ions. In addition, additives in the electroplating process also prevent the formation and growth of pyramidal structures and reduce the roughness of the coatings, having the surface be glossy [6, 9]. Similar changes in the deposit structures occur in a research by Wang, Gao, Xu, and Xue (2005). In this exploration utilizing electrodepositions, pyramidal particles and polyhedral crystals get the surface of polycrystalline nickel rougher than that of nanocrystalline nickel [10].

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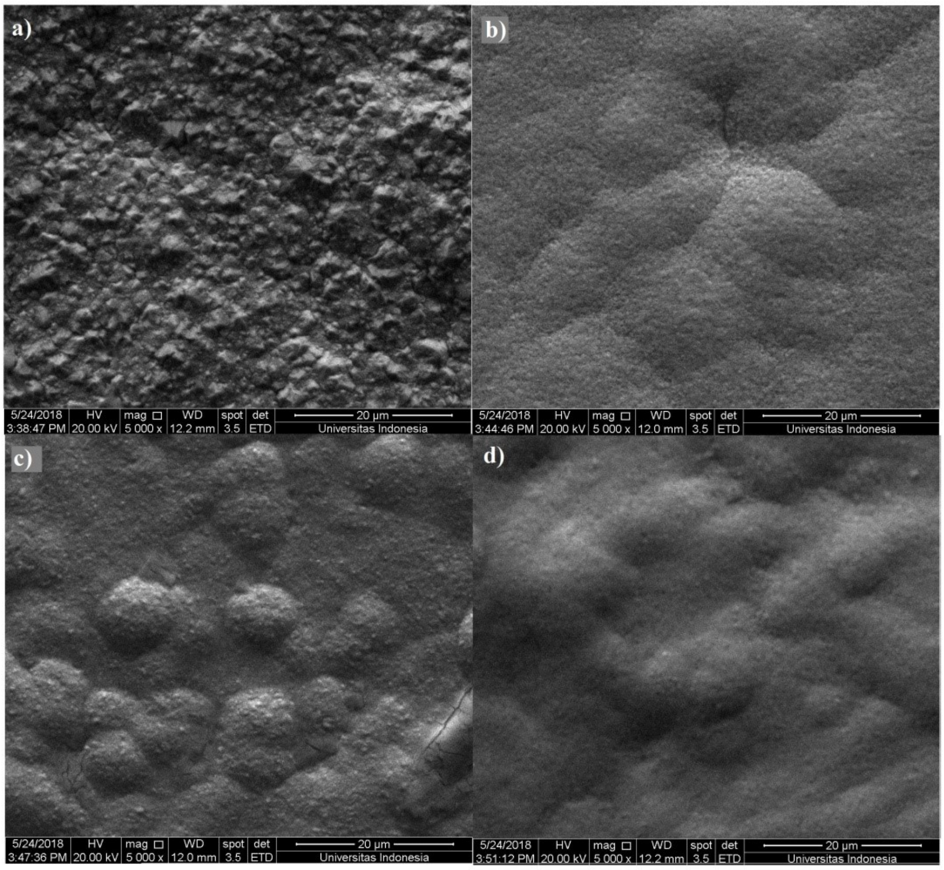

Figure 4. The micro photography of nickel-electroplated steel with saccharin addition; a) 0 g/L, b) 1 g/L, c) 3 g/L, and d) 5 g/L.

3.3. X-Ray Diffraction (XRD) Observation

Figure 5 shows the trends of deposits particles size through the XRD analysis based on saccharin concentrations. The analysis reveals that the nickel deposits have a FCC crystal structure: fields (111) and (200). The addition of saccharin during electroplating has proven to widen the peak, indicating that the grain size of the deposits becomes smaller.

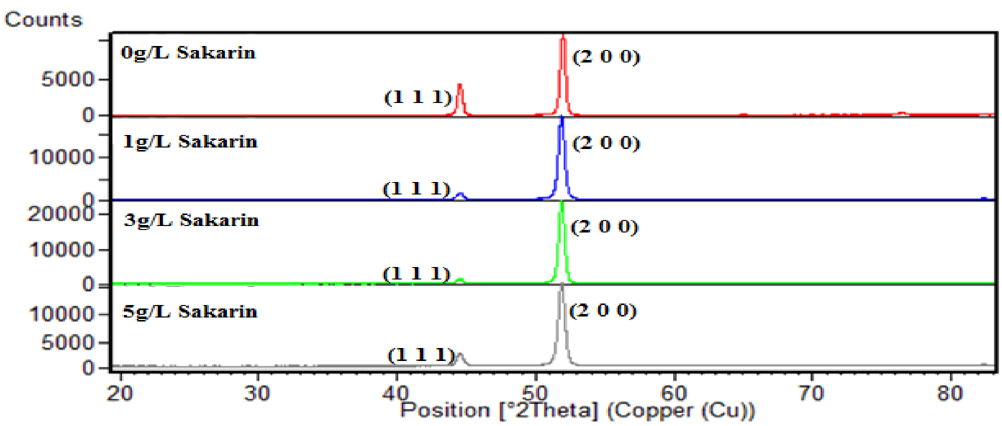

Figure 5. The trends of nickel particle size based on saccharin concentrations

Measuring nickel particle size through Scherrer equation was carried out on fields (111) and (200) because both have the highest intensity. Table 2 and figure 6 provide the details of the nickel grain size calculation based on Scherrer equation and the details of the changes in the grain size.

Table 2. The nickel grain size.

Saccharin Concentrations (g/L)	Grain Size (nm)		
	Peak/2θ		Rates
	(111)/44.5°	(200)/51.8°	
0	24.1	25.5	24.8
1	18.3	24.7	21.48
3	18.3	20.03	19.43
5	17.55	17.22	17.39

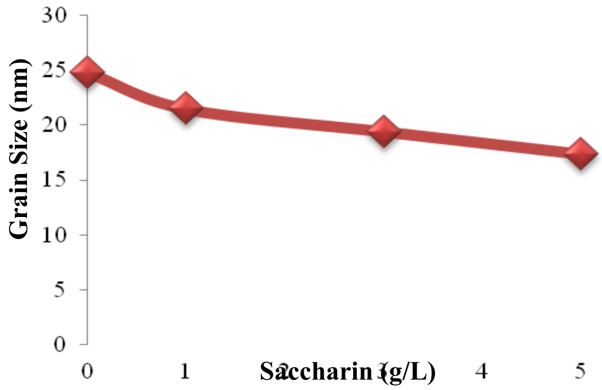

Figure 6. The effect of saccharin concentrations on the grain size.

The addition of saccharin to nickel coatings through electroplating has proven to reduce the nickel grain size. The small grain size leads to smoother layers. These smooth surfaces indicate the evenness of nickel layers. This further raises the brightness of the layers. Among all saccharin concentration variations, 5 g/L results in the smallest grain size, 17.39 nm, and the brightest light. In connection with brightness, Yli-pentti (2014) suggested that saccharin is one of the traditional brighteners widely used.

The workings of saccharin in decreasing the grain size of the nickel deposits are as follows. Saccharin blocks the surface of the substrates through the formation of complex compounds that effectively increases the frequency of nucleation, but decreases the diffusion of ions absorbed on the cathode surface. This phenomenon not only hinders the growth of nickel particle size but also affects the hydrogen evolution and distorts the cathodic overpotential [6].

4. Conclusion

The conclusion of all off the above investigations is, first, the addition of saccharin to nickel deposits through electroplating alters the deposits structure, from pyramidal to globular and colonial. The layers with the globular structure have a more even appearance than those with the pyramidal one. Secondly, the addition of 5g/L saccharin in nickel electroplating most effectively reduces the nickel grain size, from 24.8nm to 17.39nm, among all concentrations.

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