Various current and electrolyte solutions of electroplating for medium carbon steel towards coating performance as corrosive protection

V A Setyowati1*, A C Niam2, F Abdul3, and Darto1

1 Mechanical Engineering Department, Institut Teknologi Adhi Tama Surabaya, Arief Rahman Hakim 100, Surabaya (Indonesia)
2 Environmental Engineering Department, Institut Teknologi Adhi Tama Surabaya, Arief Rahman Hakim 100, Surabaya (Indonesia)
3 Materials and Metallurgical Engineering Department, Institut Teknologi Sepuluh Nopember, Keputih, Surabaya (Indonesia)

*E-mail: vuri@itats.ac.id

Abstract. Electroplating is utilized to protect metals from environmental influences. This study aims to determine the effect of solution composition and electric current on coating thickness and to observe morphology with nickel plating. The electrical currents were set in 0.5A, 1A, and 1.5A. Meanwhile, the electrolyte solutions consist of NiSO4, NiCl2, H3BO3, H2O, and were added in different compositions. In this research, several characterizations were conducted to measure the thickness of the coated layer using a micrometer. SEM (Scanning Electron Microscopy) was used to observe the surface morphology. The thickness test is shown that the higher current obtained the thicker coating layer. Moreover, the higher compositions of electrolyte solutions affect more coating layer on the base metal surface. The rough surface was depicted on metal coated in low current. New iron-nickel and iron-nickel-oxide was detected by X-Ray Diffractometer (XRD) as Fe0.64Ni0.36 and NiFe2O4.

1. Introduction

Commonly, steel are used as construction materials with various carbon composition and classified as low carbon (<0.25% C), medium (0.25–0.70% C), and high carbon steel (0.70–1.05% C). Carbon percentage contributes to different mechanical properties. Carbon steel is common materials for construction of pipeline to supply oil and gas [1]. The corrosion problems mostly occur in oil and gas related to the exposure conditions of carbon steel. Corrosion of steel or metal is an electro-chemical reaction between the metal and its environment in which the metal revert to iron oxide [2]. Corrosion is the disintegration of the engineered material into its constituent atoms due to chemical reactions with its surroundings, which also means that corrosion is an electrochemical oxidation of metals in reaction to oxidants such as oxygen [3]. The alternative method to prevent corrosion is electroplating. It is one of several methods to reduce corrosion rates and protect materials from corrosion. Electroplating is a process of electro deposition to produce an adherent, uniform, and dense coating with the aid of electric current on surface. The advantage of this process concerns to wear and corrosion resistance. The effect of nickel coating resulted has significant improvement [4].
Several previous studies about electroplating nickel coating had focused on parameters of electroplating. The corrosion behaviour of electrodeposited nickel due to cobalt addition [5]. Cr particles increase the wear resistance of Ni coating [6]. The effect of nickel plating on the damping behavior of unreinforced aluminum alloy and its composites [7]. Nickel plating generates uniform deposit, more compact, and provides better protection to substrate of steel [8]. Therefore, this study reports coating performance of medium carbon steel that electrodeposited by various current and electrolyte composition as corrosive protection. The coating thickness of coated Ni onto steel surface was measured to ensure the coating products. Morphological analysis using visual observation and SEM analysis was performed to confirm the differences appearance due to electroplating. New compounds of coated metal were examined by XRD analysis.

2. Experimental Methods

Mild steel type AISI 1045 and nickel were used as cathode material and anode material, respectively. Plate with a thickness of 5 mm was cut to the dimensions of 30mm x 50mm x 5mm. The sample surface was firstly cleaned from corrosion products by grinding and polishing. Thereafter, continue to wash using soap to ensure the clean surface. Electroplating is the main process that was carried out with a variety of currents and different media. Electroplating was carried out using electroplating with chemical composition of $\text{H}_3\text{BO}_3$, $\text{NiSO}_4$, $\text{NiCl}_2$ by changing the percentage of $\text{NiSO}_4$ and $\text{NiCl}_2$. Each experiment was repeated three times. The compositions of different solutions were detailed by Table 1.

|                        | $\text{H}_3\text{BO}_3$ (g) | $\text{NiSO}_4$ (g) | $\text{NiCl}_2$ (g) | $\text{H}_2\text{O}$ (ml) | Media |
|------------------------|-----------------------------|---------------------|---------------------|---------------------------|-------|
| Solution 1              | 40                          | 100                 | 50                  | 1000                      | Solution 1 |
| Solution 2              | 40                          | 125                 | 60                  | 1000                      | Solution 2 |
| Solution 3              | 40                          | 150                 | 70                  | 1000                      | Solution 3 |

Electroplating current was set up at 0.5 A. 1 A and 1.5 A. The electroplating time was set in 20 minutes per experiment so that AISI 1045 steel coated with nickel was obtained. The last stages were characterizations. The thickness of the electroplating Ni coating was measured using a micrometer with a precision of 0.001 mm. Data were collected at 3 points as the figure below. Visual observations were performed to determine differences in the sample before and after coating. Meanwhile, to find out more details about the coating results, observations were made using the Scanning Electron Microscope (SEM). Analysis of compounds using X-Ray Diffractometer (XRD) was carried out to determine compounds that are formed from the results of electroplating.

3. Results and Discussions

Measurement of coating thickness was carried out to determine the results obtained by using current variations and the electrolyte solution compositions. In this measurement, micrometer screw as measuring instrument was used to measure the thickness with an accuracy of up to 0.001mm. The results of layer thickness obtained are shown in Table 2.
Figure 1 denotes the higher the current used, the thicker the layer produced. Meanwhile, the variation in solution composition also shows that the more composition of the solution used, the thicker the layer obtained. The increase in thickness is insignificant as the result of the variation in current. By observing figure 1, it can be seen that there is an effect between the various currents on the resulting layer thickness and estimated using Faraday’s law [9]. This is consistent with Faraday’s law that the mass formed in the electrochemical process is directly proportional to the electric current.

Figure 1 presents that the thickness of the coating results is increase. The higher the concentration of NiSO$_4$ and NiCl$_2$, the more nickel will be available in the electrolyte. Therefore, the chance of the nickel to stick on the specimen will also be higher. In the electroplating process, the NiSO$_4$ electrolyte solution breaks down into Ni and SO$_4^{2-}$ ions. The electrolyte cation (SO$_4^{2-}$) attaches to the anode. At the cathode, the steel plate will release oxygen to the nickel solution (NiSO$_4$) due to the presence of a direct electric current with a constant voltage. As a result, nickel (Ni) ions will be deposited on the surface of the steel then steel is coated with nickel. On the other hand, at the anode, nickel (Ni) coating material binds the oxygen released by the steel. The nickel plating material will experience a bond that will dissolve in the nickel electrolyte (NiSO$_4$) has coated the plate? Thus, the nickel electrolyte solution (NiSO$_4$) will be remain stable. Consequently, the nickel (Ni) coating material will gradually decrease or run out.

**Table 2.** The layer thickness of various current and electrolyte solution for Ni-electroplating

| Current (A) | Media | Before Plating | After Plating | Thickness (mm) |
|------------|-------|----------------|---------------|----------------|
|            |       | Measurement for 3 times (mm) | Average ± Dev (N) | Measurement for 3 times (mm) | Average ± Dev (N) | P – N |
| 0.5 A      | Solution 1 | 4.81 ± 0.00305 | 4.835 ± 0.00212 | 0.022 |
|            | Solution 2 | 4.64 ± 0.0007 | 4.666 ± 0.00624 | 0.023 |
|            | Solution 3 | 4.76 ± 0.00251 | 4.788 ± 0.00251 | 0.025 |
|            | Solution 1 | 4.72 ± 0.00212 | 4.745 ± 0.00152 | 0.025 |
| 1 A        | Solution 2 | 4.751 ± 0.00152 | 4.781 ± 0.004 | 0.026 |
|            | Solution 3 | 4.64 ± 0.00351 | 4.670 ± 0.00321 | 0.032 |
|            | Solution 1 | 4.595 ± 0.0023 | 4.636 ± 0.001 | 0.043 |
| 1.5 A      | Solution 2 | 4.742 ± 0.0003 | 4.786 ± 0.002 | 0.046 |
|            | Solution 3 | 4.740 ± 0.00351 | 4.795 ± 0.004 | 0.055 |
The morphological observations were conducted by visual appearance and using a 500x magnification of SEM. This observation was carried out to determine the difference of the electroplating using variations in current and electrolyte solution composition in mild steel AISI 1045. A high current of electroplating causes electrons more reactive. It will generate the portion of the accumulated movement of electrons and the displacement of the material at the two electrodes gets bigger. The impurities inside the anode will also be transported away, so the result will be darker. In the other case, a low concentration of electrolyte composition generates a darker surface appearance.

According to the results of plating using a variation of the current strength of 0.5A in Figure 2. The less composition of the solution used, the black plating results are obtained on the outer side of the plating. It can be shown in Figure 2 and the possible reason is the efficiency of the process where the conditions have not met the ideal conditions. For example, the distribution of the electric current needed to generate the movement of electrons at the two electrodes (specimen and anode), including the electron transfer of material is transported by electrically charged ions through the solution.
In Figure 3, it was clear the condition of the coated and uncoated parts. On the uncoated side, the surface looks flat, while the coated material shows the presence of adhered coating grains. SEM image of 2 (1A) sample shows there is a leak in the tape which should have been tightly closed during electroplating. So, there are electrodes attached to the part of the tape. This can be obviously seen in the SEM images that particulates are not evenly distributed and spread on the part. It is covered by the tape and should be clean like the other photos. In the SEM image of 1 (0.5A) sample, there is a layer shape with the particulate indicating that is tidier, evenly distributed, and there is a little gap. The gap is also clearly visible in the SEM image of 1 (1.5). The empty gaps also occur on the coated material. The higher current density is used for electroplating, it can cause the influence of the condition of the coating layer particles. Low current generates more particles that are not melted. Therefore, the binding power of the reinforcing particles is getting low. The low bond strength will result in a low value of coating hardness and thickness. This condition also causes the empty space between the particles to form porosity. Consequently, the corrosion rate obtained is higher. Meanwhile, the SEM images denote a difference in the size of the coating grains in the electroplating process. For 0.5A of current, it has a larger layer particulate. This can occurred because the layer that is attached and darker. Larger grain size of the substrate, the resistance of electrons to penetrate through boundary will decrease the resistance of corrosive attack [10]. The surface compaction improvement and refinement of grain size not only enhance mechanical properties but also important to increase corrosion resistance [11]. Regarding to SEM image it has different surface roughness and absolutely correlate to corrosion resistance [9].
Figure 3. SEM image samples after coated using various current and media of electroplating

Figures 4 presents the XRD result after electroplating, the Fe_{0.64}Ni_{0.36} alloy phase (JCPDS card no 47-1405), and NiFe_{2}O_{4} oxides (JCPDS card no 54-0964) was appeared [12]. There is no shift peak for all various samples and slight peaks of XRD pattern indicates crystalline structure [13]. The XRD image in the figure shows the presence of a new compound formed in the coating product. The most dominant compound is Fe_{0.64}Ni_{0.36}. There is a NiFe_{2}O_{4} compound when coated with a current of 0.5A and 1A but no current conditions of 1.5A are visible. The opposite condition occurs when using electrolytic media with solution composition 2. The dominant compound is Fe_{0.64}Ni_{0.36} for the three variations of the current, but the NiFe_{2}O_{4} compound is only formed when the current used during electroplating is 1.5A. A different thing happens in electrolyte 3 where the metal coated with a current of 1A is formed by NiFe_{2}O_{4} and Fe_{0.64}Ni_{0.36} compounds, while in other variations only Fe_{0.64}Ni_{0.36} compounds are formed. Peak at position 2θ of 76.89° in figures 4 with the composition of solutions 2 and 3 shows intensity enhancement. This phenomenon is influenced by the high of the ampere used. Meanwhile, compositions
of solution 1 looks inversely proportional. It could be occurred because of the low concentration so that the Ni dissolved less.

Figure 4. XRD pattern of coated samples using various current in (a) electrolyte solution 1 (b) electrolyte solution 2 (c) electrolyte solution 3

4. Conclusion

The composition of the electrolyte solution and the current in the electroplating of AISI 1045 steel have a significant impact on the layer thickness analysis, morphological analysis, and compound analysis. In
the thickness test, it can be shown that the higher current, the thicker coating layer was gotten and the higher the solution composition, the thicker the coating results. From the morphological observations, it can be concluded that the electroplating layer granules formed at a current of 0.5A are greater than those found at 1A and 1.5A currents. Fe_{0.64}Ni_{0.36} and NiFe_2O_4 compounds are formed on the metal-coated by Nickel.

5. References

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