Comparison of Different Surface Pre-Treatment on Mild Steel for Cobalt-Nickel-Iron Electroplating

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ABSTRACT

Electroplating is extensively practiced in the industry to fabricate corrosion-protective coatings for steel in large-scale production. Mild steel easily rusts at ambient temperature thus surface pre-treatment is mandatory to eliminate rust and superficial scale from the steel. Pre-treatment ensures that the steel surface is free from contaminants, which may interfere with the surface quality of the protective coating. This research is done to investigate the effect of different pre-treatment methods on the surface quality of mild steel rings and cobalt-nickel-iron coated mild steel rings. These surfaces were achieved by polishing the ring and subjected to alkaline degreasing, followed by immersion in sulfuric acid or sodium chloride at 10 vol % concentration and different immersion times (50 s, 55 s, and 60 s). Direct electroplating was applied to fabricate the cobalt-nickel-iron coating. The surface morphology of metal substrate and coating after electroplating were tested by scanning electron microscope (SEM), energy dispersive X-Ray (EDS), Vickers hardness, and surface roughness tests. Both types of pre-treatments provided lower surface roughness on the metal substrate and resulted in full coatings without voids formation. The results showed that pre-treatment using sulfuric acid exhibited higher hardness and a smoother coating surface. Agglomerates and cracking were observed on the surface coating treated with sodium chloride.

Introduction

Preventing corrosion in ferrous metals is a major concern among engineers. Corrosion can happen at all stages in manufacturing, finishing, and maintenance. The most common forms of corrosion protection are galvanization [1], alloying [2], cathodic sacrificial protection [3], and barrier coating [4]. Recent trends in coating protection have shifted focus to nontoxic options to replace chromate and phosphate conversion coating [5]. Cobalt and its alloy coating [6] are reported to have good corrosion inhibition. Besides, green fabrication of zinc-cobalt alloy coating improved the corrosion resistance by up to 27.6% [7]. The surface hardness, wear, and corrosion resistance were enhanced with the presence of cobalt coating in nanocrystalline size [8]. However, the corrosion resistance level of the coating depends on the quality of the coating itself and the surface of the substrate to be coated. Surface quality is important as the material surface directly contacts with external loads or forces such as friction, wear, corrosion, and fatigue that may affect the performance of the materials. Surface coating is incorporated on the metal surface to improve the lifetime of the metal.

There are many methods to fabricate coating as a protection layer on ferrous metals such as hot dip galvanizing, thermal spraying, chemical vapor deposition, laser cladding, and others. Among the techniques for coating synthesis, direct electroplating is of great interest for industrial use [8]. This technique lies in its versatility, moderate costs, large-scale production, and easy control thickness. In this technique, metal substrate surface pre-treatment is recommended prior to the electroplating process. The surface pre-treatment is usually associated with sandpaper polishing, cleaning, pickling, activation, or passivation to prepare the material for plating [9]. It is reported that surface pre-treatment could improve the wettability of the surface, which allowed for a better cohesion of the coating layer and ensured a homogeneous coating [10]. Besides, the surface pre-treatment helped to remove salts, soaps, or alkaline cleaning products left on the surface to avoid staining, skip plating, or even delamination and blistering.

The most common pickling used in mild steel surface treatment is corrosive fluid such as hydrochloride acid [11]. Another corrosive acid used in pickling could be sulfuric acid [12], or a mixture of acids with toxic inhibitors [13]-[14]. Alkaline such as ammonia fluoride was also proposed in the pre-treatment of mild steel. A neutral solution such as sodium chloride was incorporated into acid, which removed the oxide layer from the substrate surface and make the surface bright without weight loss [15]. Sodium chloride has been shown to have a beneficial effect in the pickling of mild steel if mixed

with sulfuric acid instead of the addition of hydrochloric acid into sulfuric acid [16]. The addition of sodium chloride enhanced the scale removal rate. It also figures depicted that the corrosive factor of the solution decreased if the concentration of the sodium chloride was increased up to 10 vol% [17] because of the oxygen solubility in water [18].

The surface profile after surface pre-treatment of steel also showed a significant effect on coating and corrosion protection for long-term service life. A thicker coating could be adhered to by a rough metal substrate to give better corrosion protection, but there is a limit [19]. A very rough surface might contain contaminant debris and a deep valley that is difficult to be coated. It could give an impact on the pitting corrosion [17]. Thus, the surface profile investigation is essential to ensure the quality of the coating.

The above-mentioned previous study had shown that the key to having a quality final coating from the electroplating process depends on the type and concentration of the pickling solution for substrate surface pre-treatment. The objective of this study is to investigate the role of sodium chloride and sulfuric acid separately on mild steel surface pre-treatment. The surface profile of mild steel before and after electroplating with a cobalt-nickel-iron corrosion protection layer was also reported in this study.

Experimental Work

Mild steel is the most widespread form of steel in the world. Mild steel rings were used as substrates, with a total surface area of 10.79 cm^2. The mild steel ring specimens contained 0.15% of carbon and more than 98% of iron. The specimens were polished with silicon carbide sandpapers of 240, 400, 600, and 1000 grits using a Buehler Handimet 2 Roll Grinder. After that, the specimens were cleaned using sodium hydroxide alkaline degreaser for 10 minutes at 65 °C. The alkaline degreasing was used to remove water-insoluble contaminants. The specimens were then rinsed with distilled water and prepared for the pickling process. Pickling provides deep cleaning while also activating the metal surface by removing the oxide layer from the surface of the material. The pickling process has resulted in a pure metal surface, which is free of contaminants and oxides [14]. Subsequently, the specimens were immersed in different solutions for varying periods, as shown in Table 1.

Table 1: Pickling solution and immersion time in mild steel ring substrate

Type of pickling solution	Time of immersion (seconds)		
Sulfuric acid	50	55	60
Sodium chloride	50	55	60

A former study proposed using 12.5% of sulfuric acid and immersion time ranging from 35 s to 55 s [12]. It is recommended that 10% to 14% sulfuric acid be used for carbon steel containing less than 0.4% carbon. Therefore, in this study, 10 vol% sulfuric acid with prolonged immersion time ranging from 50 s to 60 s was applied. For comparison purposes, 10 vol% sodium chloride was also used. The specimens were then washed with distilled water and air-dried.

In the electroplating process, the plating solution was prepared in a sulfate bath electrolyte with the presence of cobalt sulfate, nickel sulfate, iron sulfate, and some additives as tabulated in Table 2. The raw materials shown in Table 2 were mixed to produce a 500 ml volume of sulphate bath electrolyte. The electrolyte was prepared at a pH level of about 3. The temperature throughout the whole electroplating process was kept around 50 °C to 54 °C. While the acidic sulphate bath and operating temperature were determined based on parameter optimization reported in a previous study [21].

Table 2: Composition of sulphate plating solution (per 500 ml solution)

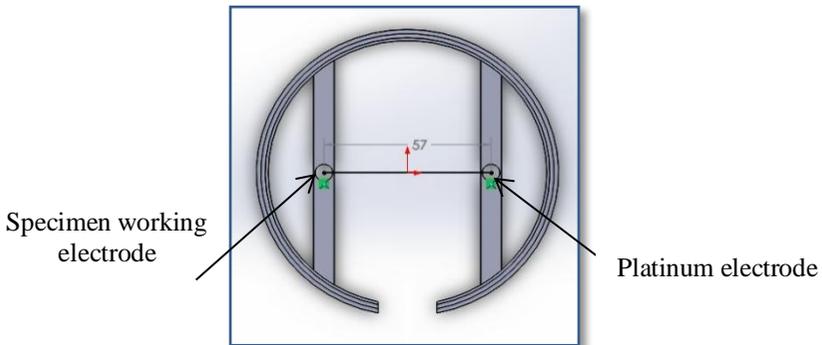| Composition	Amount (gram)
Cobalt sulphate	7.03
Nickel sulphate	17.52
Iron (II) sulphate	2.78
Boric Acid (additives)	8.24
Ascorbic Acid (additives)	5.87

Figure 1: Schematic diagram of electrodes holder in electroplating process (top view)

The electrodes used in the electroplating process were clipped to the plastic-designed holder, as presented in Figure 1. A platinum plate was used as the auxiliary electrode (anode), whereas the mild steel ring was acting as

the working electrode (cathode). Both electrodes were placed at a distance of 57 mm to stabilize the ions' transfer during electroplating. Mild agitation was conducted throughout the electroplating process to avoid bubbles accumulating on the cathode surface, which may cause hydrogen evolution. A constant current of 1.0 A was applied for 20 minutes of electroplating time. A resultant mono-layer of cobalt-nickel-iron was produced on the mild steel specimen after electroplating.

The quality of the surface substrate and surface coating was evaluated by measuring their surface roughness and surface hardness. The surface roughness of the coating was measured using the Mitutoyo surface measuring instrument (Surftest SJ-410) following the ISO1977 standard. Three randomly selected points on the flat side of each specimen were used to measure the surface roughness. At each point, the probe travel was set at a speed of 0.5 mm/s and a travel length of 4.8 mm. The most common expressions for surface roughness are *Ra* and *Rq*. *Ra* is the arithmetic mean of absolute values of the evaluation profile deviations from the mean line, as shown in Equation (1). *Rq* is referred to as the square root of mean roughness, as expressed in Equation (2) [19].

$$R_a = \frac{1}{n}\sum_{i=1}^{n}|Yi| \tag{1}$$

$$R_q = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(Yi)^2} \tag{2}$$

where *n* is the number of segments over the evaluation length and *Yi* is the deviation from the mean line.

In terms of surface hardness, the method of measurement was using the Mitutoyo Vickers hardness test. The loading (*F*) applied in the hardness test was 0.5 kilograms-force. Similarly to the surface roughness test, the surface hardness was tested at three different areas of specimens, while avoiding the defect area on the surface. The Vickers hardness was determined using Equation (3) [20], where diagonal lengths of the indentation (*D*) left on the surface of the material were measured.

$$\text{Vickers hardness, } HV \approx 1.854\frac{F}{D^2} \tag{3}$$

where *F* is the load in kgf, and *D* is the arithmetic mean of the two diagonal lengths.

The pre-treated substrate surface and surface coating morphology were examined using a Hitachi TM3030 PLUS model scanning electron microscope

(SEM) attached with an energy dispersive X-ray (EDS) machine. The specimens were mounted on aluminium stubs, using carbon double-sided tapes for SEM and EDS analysis. Cross-sections of samples were prepared to determine the coating thickness and elemental composition.

Results and Discussion

Surface roughness

Figure 2 displays the line profiles for metal substrates before and after surface pre-treatment. It can be seen in Figure 2(a) that the metal surface without pre-treatment is rougher due to large deviations from the mean line. The average surface roughness, *Ra* was recorded at 1.536 μm and *Rq* at 2.616 μm. Figure 2(b) demonstrates the surface of the metal surface after pre-treated with sulfuric acid. The surface became smoother and showed a *Ra* of 0.327 μm and *Rq* of 0.480 μm. The drastic drops of *Ra* and *Rq* after pre-treatment revealed that the original mild steel had more defects such as contaminants, oxides, valleys, and grooves. A similar finding was found in contaminated Ni-Co substrate pre-treated with sulfuric acid [12]. The burrs or oxides on the substrate surface were completely removed by modified pre-treatment with sulfuric acid and further enhanced the adhesion of the coating [19]. It was believed that the oxide and contaminants interfered with the adhesion of the coating and caused corrosion creep, where the corrosion occurred under the coating interfaced with the metal surface [19]. Thus, it was essential to remove the contaminants of the metal surface to avoid corrosion failure on the coated metal.
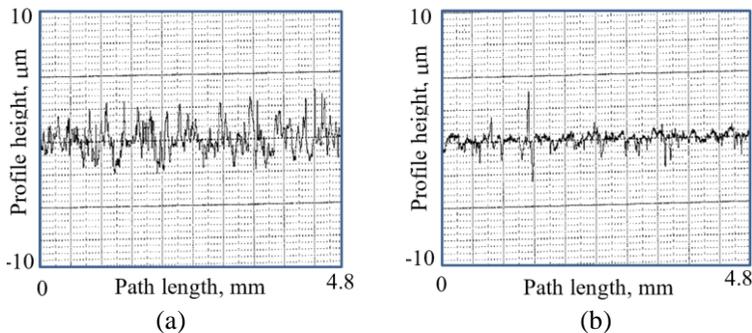

Figure 2: Roughness line profiles for mild steel surface substrate; (a) original, and (b) pre-treated with sulfuric acid at 60 seconds of immersion

Table 3 depicted the results of the surface roughness of mild steel substrate after being pre-treated with different types of solution. It is clearly

shown that the average surface roughness, *Ra* was significantly reduced in sulfuric acid solution as the immersion time increased. However, the surface roughness, *Ra* of the mild steel substrate was not affected by the time of immersion in sodium chloride. The roughness after surface pre-treatment had improved compared to the original metal surface (*Ra* of 1.836 μm) without pre-treatment. The average *Rq* was lower in acid solution indicating that the deviations between peaks and valleys were smaller. When sulfuric acid (H_2SO_4) reacted with steel, the iron oxide on the mild steel (mainly iron element) surface was chemically removed and transformed into iron sulphate [12]. The reaction of cations and anions between sulfuric acid and steel is depicted in Equation (4).

Table 3: Surface roughness of mild steel substrate after surface pre-treatment

Pickling solution	Immersion time, t (s)	Average surface roughness, *Ra* (μm)	Average square root of the arithmetic mean, *Rq* (μm)
No pre-treatment (reference)	0	1.536	2.616
Sulfuric acid	50	0.927	0.502
	55	0.573	0.531
	60	0.327	0.480
Sodium chloride	50	0.652	0.671
	55	0.641	0.808
	60	0.693	0.782

The iron sulfate ($FeSO_4$) was believed to adhere to the steel surface and formed a protective layer [22] that covered the grooves and valleys, hence giving a lower value of *Rq*. However, the $FeSO_4$ layer was easily scaled off and poorly adherent. The $FeSO_4$ layer could not permanently protect the base mild steel from rusting. Thus, a permanent coating is essential to overcome the problem. Furthermore, the chloride ions in sodium chloride (neutral solution) would reduce metal loss but could initiate the formation of pits and may lead to localized corrosion, which is also known as pitting corrosion [17].

$$H_2SO_4 + Fe \rightarrow FeSO_4 + H_2 \qquad\qquad (4)$$

Figure 3 compared the surface roughness of cobalt-nickel-iron coated mild steel after electroplating. The surface coating of the specimen pre-treated with sodium chloride is rougher compared to the surface coating pre-treated with sulfuric acid. The surface roughness dropped when the immersion time increased. There was about 55% of reduction in surface roughness when the specimen was immersed in acidic solution from 50 seconds to 60 seconds.

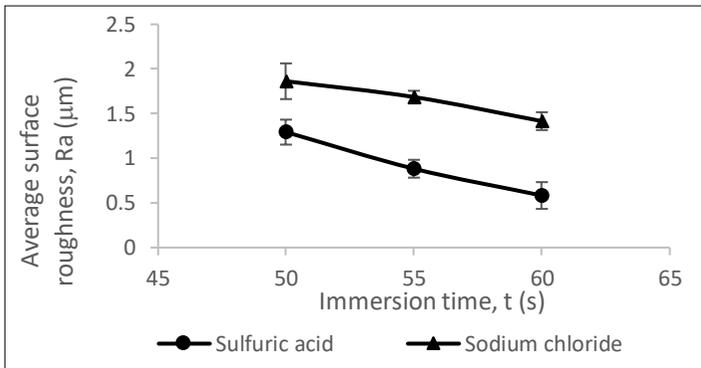However, the reduction for specimens treated with sodium chloride was smaller (24%).

Figure 3: Surface roughness of surface coating pre-treated with different pickling solutions

Surface morphology

Figure 4 represented the surface morphology of metal substrates. The results indicated that surface pre-treatment had a significant effect on roughness and coating quality. The results show good agreement with *Ra* values as tabulated in Table 3. The metal substrate surface without treatment is the roughest. There are many valleys found in Figure 4(a), resulting in higher *Rq* values due to high deviations from mean roughness values. In contrast, Figure 4(b) showed that the metal surface pre-treated with sulfuric acid appears smoother, with only a little debris found on the surface. A similar finding had been reported [23], where static pickling produced a smoother surface than dynamic pickling.

In terms of the effect of sodium chloride pickling solution, small voids can be seen scattered throughout the surface of the specimen, as revealed in Figure 4(c). The formation of these voids could be due to corrosion activity on the surface of base metal when treated with sodium chloride [17]. It is known that chloride ions in sodium chloride promoted the hydration of iron ions from the mild steel substrate. Sodium chloride is the corrosion media that helps to remove the iron ions accumulated at the surface. It is reported that the chloride (Cl) ions are incorporated with iron (Fe) ions to form an iron oxide layer and leave cationic vacancies, which then condense at the irons interface to form small voids. These small voids are also known as pits [24]. The formation of the pits upon pre-treated with sodium chloride was illustrated in Figure 5. Figure 4(d) showed the cross-section of the specimen pre-treated with sodium chloride. It can be seen that pits are formed at the surface of the base metal. A similar SEM cross-sectional morphology of the pit was reported in the low-carbon steel treated with chloride solution [25].
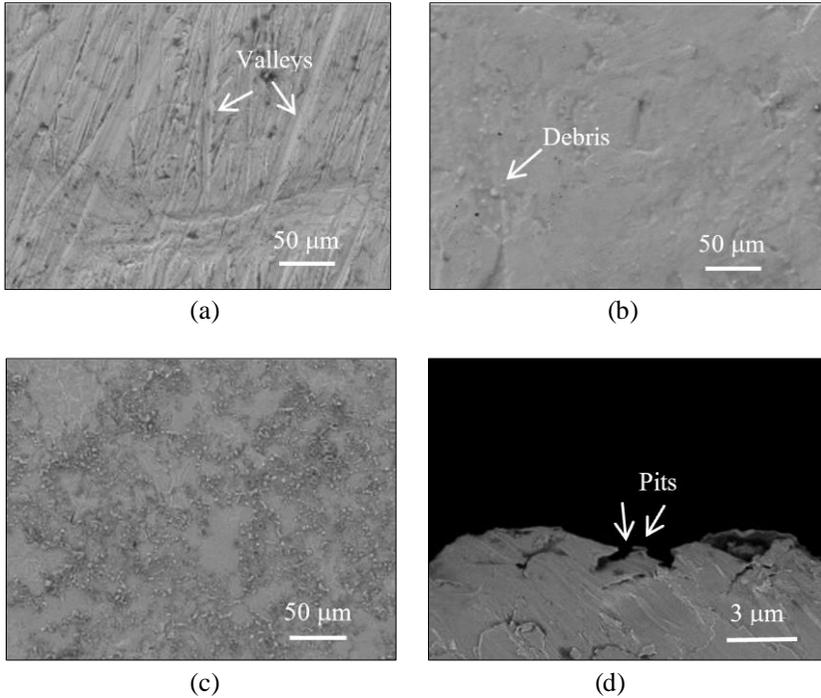
Figure 4: SEM images; (a) original mild steel, (b) mild steel pre-treated with sulfuric acid at 60 s, (c) mild steel pre-treated with sodium chloride at 60 s, and (d) cross-section of mild steel pre-treated with sodium chloride at 60 s

The surface coating with and without pre-treatment were presented in Figure 6. Figure 6(a) showed numerous agglomerates and voids appearing on the surface coating of metal specimens without surface pre-treatment. The rough surface of the coating contributed to the highest *Ra* values of 2.156 µm due to the large deviation of peaks and valleys from the mean line over time. The substrate surface pre-treated with sulfuric acid shows a homogeneous coating without voids and agglomerates. The coating surface morphology on the sample pre-treated with sulfuric acid was shown in Figure 6(b). The result is consistent with the surface roughness measurement, which indicated the lowest value. The uneven surface of the metal substrate is susceptible to uneven coating formation and thus results in higher surface roughness. In contrast, it can be said that the substrate surface pre-treated with sulfuric acid showed a homogeneous coating without voids and agglomerates and gave the lowest surface roughness as shown in Figure 6(b).
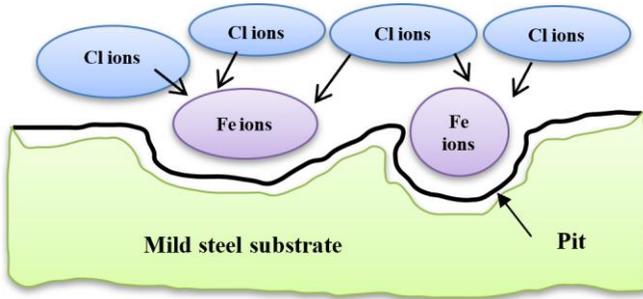
Figure 5: Illustration of sodium chloride reacted with mild steel substrate
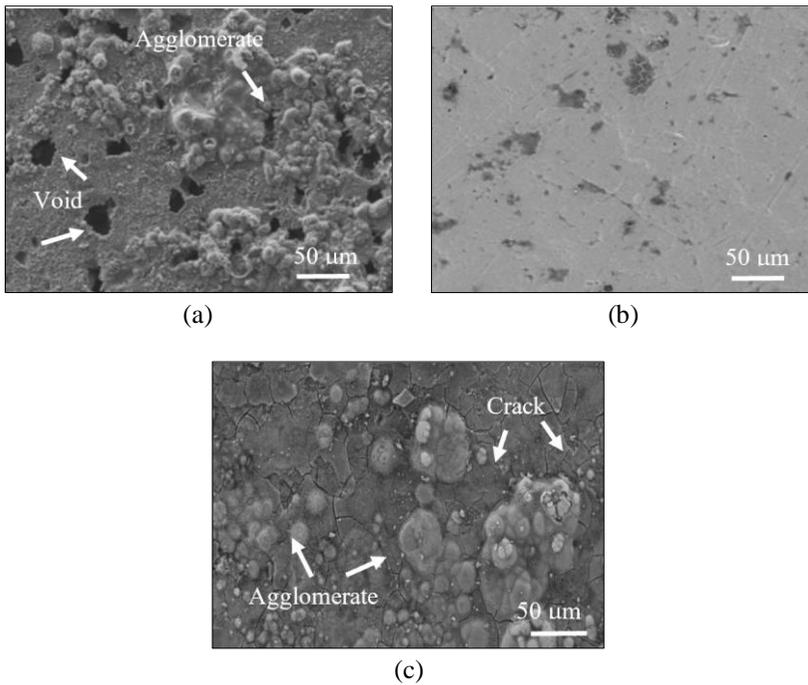

(a)

(b)

(c)

Figure 6: SEM images; (a) coating without pre-treatment, (b) coating pre-treated with sulfuric acid at 60 s, and (c) coating pre-treated with sodium chloride at 60 s

Figure 6(c) demonstrated that the coating fully covered the pits on the surface of the specimen pre-treated with sodium chloride, which led to the formation of agglomerates. This is due to surface irregularities on the pre-treated specimen as shown in Figure 4(c). Moreover, cracks were also found

throughout the entire surface. This defect was probably caused by agglomeration, which exhibited brittle rupture [26]. As a result, the surface pre-treated with sodium chloride had become rougher after being electroplated with cobalt-nickel iron.

Figure 7 showed a comparison of the thickness of the coating formed with different pre-treatment. The thickness of a mono-layer of cobalt-nickel-iron coating ranged from 10 μm to 15 μm at a constant electroplating time of 20 minutes. The thickness of the coating did not vary too much. In fact, the main factor influencing coating thickness is the electroplating process parameters [12]. The surface coating shown in Figure 7(a) is smooth on the specimen pre-treated with sulfuric acid. It is evident from Figure 7(b) that the Co-Ni-Fe coating was rougher on the specimen pre-treated with sodium chloride. Overall, the roughness of the coating surface is much lower than the thickness of the coating, which is suitable for automotive coatings that require smooth and low-profiled surfaces [19].
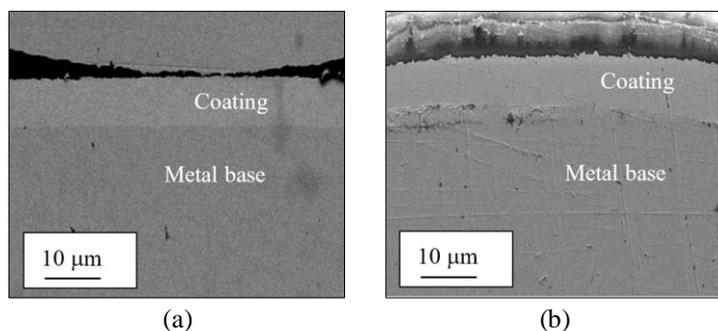

| (a) | (b) |

Figure 7: SEM cross-section images coated mild steel ring pre-treated with; (a) sulfuric acid, and (b) sodium chloride at 60 s time immersion

The EDS analysis was performed on the coating as well as the metal base for both pickling solutions. Both solutions presented similar results where no contaminant was visible on the coating and metal base as shown in Figure 8. It is found that the coating showed elements of cobalt (Co), iron (Fe), nickel (Ni), and carbon (C). The metal base is purely Fe and carbon (C). This result shows that all pickling solutions and contaminants were removed successfully after pre-treatment prior to Co-Ni-Fe electroplating.
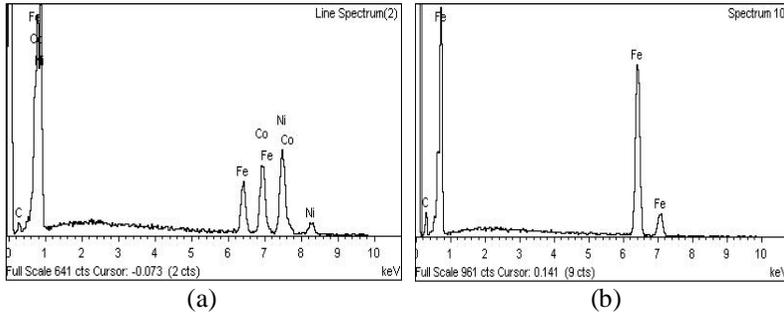
(a)   (b)

Figure 8: EDS spectrum; (a) Co-Ni-Fe coating, and (b) metal base

Surface hardness

Figure 9 showed the average surface hardness for coated specimens pre-treated with multiple pickling solutions. The sulfuric acid solution had shown to produce greater hardness compared to the sodium chloride solution. Thus, the addition of coating on the metal substrate should increase its hardness. However, it is found that the coated specimen treated with sodium chloride showed similar hardness to the mild steel substrate (114 HV). It is believed that the pits formed on the substrate surface weakened the bonding between the coating and substrate. The coating did not function well in protecting and enhancing the hardness due to the film-breaking mechanism caused by pits [24]. Cracks also occurred on the surface of the specimen pre-treated with sodium chloride, which further weakens the resistance of the specimen to external force.
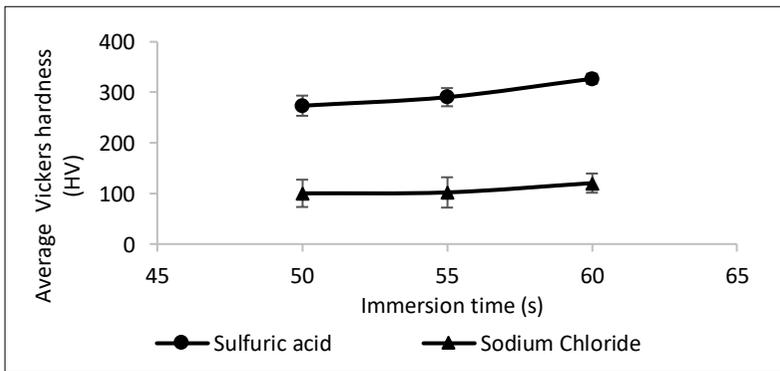

Figure 9: Surface hardness of coated mild steel pre-treated with multiple pickling solutions

The finding in Figure 9 correlated well with the surface roughness. A lower surface roughness resulted in better hardness. Hardness measured the material's resistance to plastic deformation. The acidic solution provided a smoother surface for coating and thus reduced coating defects. The smooth substrate surface enhanced the adhesion of the coating and resulted in better resistance to plastic deformation. It is reported that material with higher hardness could be more wear-resistant [27].

Conclusion

The comparison was made between the surfaces pre-treatment of mild steel using different pickling solutions after grinding and alkali degreasing. Both types of solutions were effective in removing the contaminants, rust oxide layer as well as improving the surface profile of the initial mild steel substrate. The surface pre-treatment not only reduced the roughness of the initial substrate but also minimized the voids formation problem in the coating during electroplating. However, randomly distributed pits were observed on the surface pre-treated with sodium chloride. The surface hardness of the coating was not improved after being pre-treated with sodium chloride due to agglomerates and film cracking. In comparison, the surface pre-treatment using sulfuric acid had shown promising results at a 10 vol% concentration of sulfuric acid and 60 seconds of immersion. As a uniform coating with better hardness without peeling, pitting, and cracking was obtained, it is recommended that the surface pre-treatment process must be carefully controlled when using a corrosive solution. The intention should be to clean and activate the substrate surface but not to further corrode the steel.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

Funding

Conflict of Interests

All authors declare that they have no conflicts of interest.

Acknowledgment

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