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Surface Properties of Ternary Alloys Synthesised with Different Electroplating Parameters

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Abstract

Stainless steel 304 (SS304) is one of the most widely used steel fasteners in both commercial and industrial sectors. However, stainless steel is susceptible to failure in the harsh corrosive environment despite its good corrosion resistance. Nowadays, the electrodeposition process is gaining traction as a flexible method to improve surface properties using various types of alloys. In this study, Co-Ni-Fe alloy was chosen as a protective coating for SS304 bolts. First, deposition times of 15, 30, and 45 minutes and current densities of 28, 35, and 42 mA/cm^2 were shortlisted as variable plating parameters. The electroplated samples were characterised in terms of surface roughness, microhardness, surface morphology and surface composition. The relationship between these properties and plating parameters (deposition time and current density) was explored. In most cases, as deposition time increased, surface roughness decreased whereas microhardness increased. Besides, the higher the current density, the higher the microhardness. On the other hand, surface roughness increased up to a certain current density before dropping back.

Discipline: Material Science, Mechanical Engineering, Industrial Technology

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1 Introduction

A fastening system is crucial to any assembly or structure. Corrosion is one of the major factors that contribute to this system failure. Corrosion has been incurring a significant negative impact on society, the environment and the economy (Finšgar & Jackson, 2014). In the US alone, it cost 3.1% of the national GDP (Koch, 2017). Stainless steel 304 (SS304) is one of the widely used steels in both commercial and industries. However, stainless steel remains prone to corrosion but this can be mitigated by coatings application. A coating is a layer applied to a surface with the purpose to protect a substrate from corrosion. One of viable coating methods is electrodeposition. The lifetime of metals exposed to corrosive environments can be

prolonged by using this method which is also low-cost (Hyie, Resali, & Abdullah, 2012). It is a type of electroplating process whereby a solid, adherent and uniform coating is produced. Metal or alloy is deposited onto an electrically conductive surface by an electrolysis process from a formulated electrolyte (Lowenheim, 2014), which can be a solution made up of a simple salt or a complex salt type.

Among the various type of alloy coating, the cobalt-based alloy is a promising application for corrosion protection. Co-Ni-Fe (cobalt-nickel-iron) alloy is even better in terms of corrosion resistance than its pure cobalt counterpart (Resali, Hyie, Berhan, Salleh, & Kasolang, 2013). On top of that, Co-Ni-Fe coating demonstrated desirable wear resistance, thermal stability and surface hardness (Koay et al., 2017, Hyie, Zabri, Nik Roseley, & Nik Mohd Masdek, 2016). Co-Ni-Fe ternary alloy coating is a prospect to become an alternative for other corrosion protection that has a harmful effect on the environment like chromium coating.

It is known that plating parameters such as current density and deposition time influence the properties of Co-Ni-Fe such as thickness, morphology, hardness and corrosion performance (Resali et al., 2013, Koay et al., 2017). By using a pre-determined current density and deposition time, multiple coatings with different properties can be synthesised. However, only one combination of plating parameters will have the best corrosion. To confirm this relationship and the hypothesis that only one combination is feasible, this study attempted to synthesise Co-Ni-Fe alloy onto SS304 bolt through an electrodeposition process using different parameters which are deposition times and current density.

Since each plating parameter has different surface properties, all possible combinations of plating parameters were characterised. The synthesised coatings were scanned with an optical 3D metrology system to study their surface profile and microhardness is evaluated using a Vickers indenter. Scanning electron microscope provided supplementary information on surface morphology and composition.

2 Methodology

2.1 Materials

Stainless steel 304 bolts were selected as the substrates for this research. The other designation code for this material is UNS S30400 of the unified numbering system. The bolts were full thread heavy duty hex bolts. The dimension was 1/2"-13 x 2" UNC.

2.2 Preparation of Substrate

Ultrasonic cleaning is a surface treatment method chosen to prepare the substrate's surface. The substrate (SS304 bolt) was immersed completely in the distilled water tank of ultrasonic cleaner. Once the cleaning process was initiated, the water was heated to approximately 40°C and vibrated at 42 kHz ultrasonic frequency to remove contaminants such as dirt or dust on the surface of the substrate. The duration of cleaning was set to 5 minutes. After the substrate was taken out from the cleaner, it was left to dry naturally.

2.3 Preparation of Electrolyte

The electrolyte was made from cobalt (II) sulfate-7-hydrate, nickel (II) sulfate-7-hydrate, and iron (II) sulfate-7-hydrate. Boric acid, ascorbic acid and saccharin were used as additives. These compounds were mixed with 2 litres of distilled water. Then, the mixture was stirred and heated to 50°C simultaneously using a hotplate stirrer to dissolve the chemical compounds until the solution become clear.

2.4 Electrodeposition Process

A DC power supply that is capable of providing 12 V and 5 A of variable voltage and current was used. Two pieces of 10 cm x 10 cm platinized titanium mesh is connected in parallel to the positive terminal whereas the substrate was connected to the negative terminal to become anodes and cathode respectively. The electrolyte solution was filled in a glass tank of cuboid shape that was placed on top of a hotplate stirrer which

heats the solution and maintains it at 60 ± 5 °C. The variation of deposition time and current density were the subjects of the surface profile study because controlling these parameters were manageable compared to the others. The selected deposition times are 15, 30 and 45 minutes and the current densities are 28, 35 and 42 mA/cm^2. Nine samples were produced using a set of fixed parameters with a tweaked deposition time and current density. Different combinations in Table 1 will give different properties such as particle arrangement that influence the surface profile and other properties too. Samples were labelled using T_{XX}-CD_{YY} where T is deposition time and CD is the current density. For instance, sample T15-CD28 was synthesised for 15 minutes of deposition time and 28 mA/cm^2 current density.

Table 1: Designation code for a different combination of deposition time and current density.

Designation code	Deposition time (minutes)	Current density (mA/cm2)
T15-CD28	15	28
T15-CD35		35
T15-CD42		42
T30-CD28	30	28
T30-CD35		35
T30-CD42		42
T45-CD28	45	28
T45-CD35		35
T45-CD42		42

2.5 Surface Roughness

The surface roughness of a coating depends on the current density, deposition time, pH and agitation (Tabakovic, Gong, Riemer, & Kautzky, 2014). A 3D surface map of the head of the bolt was generated by using Alicona 3D surface metrology system. Before being placed on the measuring platform, the bolt was mated with a nut acting as a support; so the axis will be perpendicular to the surface of the platform. The measurement of the bolt was taken on the surface of the top of the head. The output parameters for this test were arithmetical mean height, Sa.

2.6 Microhardness

Vickers microhardness testing was carried out using MITUTOYO MVK-H1. The samples were secured to a clamp on the platform on both ends to ensure a flat surface. 500 gf of the load was applied for 15 seconds. 500 gf was selected to ensure the depth of indention was less than 1 µm (Resali et al., 2013). Five measurements from various locations on the surface were taken for each sample. The data were used to obtain the mean of HV value and standard deviation.

2.7 Surface Morphology and Composition

A scanning electron microscope (SEM) was used to examine the surface morphology of the deposition of nanocrystalline by using a secondary electron. A sample was placed in the vacuum chamber and 15 kV of the electron beam was focused over the sample to scan it. The sample was magnified by 1000 times to obtain a micrograph. Based on the micrograph, defects in the sample were identified. Besides, the composition of materials was obtained by Energy Dispersive X-ray (EDX).

3 Results and Discussion

3.1 Surface Roughness

The surface roughness, S_a of the SS304 substrates was 0.30865 ± 0.05043 µm. Table 2 shows the S_a values of Co-Ni-Fe coatings synthesised using different parameters. In order to simplify, the designation code is T_{XX}-CD_{YY} where T is deposition time and CD is the current density. The S_a value sample T15-CD35 had a maximum roughness of 0.95701 µm whereas T30-CD42 had a minimum roughness of 0.39498 µm.

Table 2: Surface roughness of Co-Ni-Fe coating.

		Surface roughness, Sa (µm)		
Deposition time (minutes)		15	30	45
Current density (mA/cm2)	28	0.75812	0.53851	0.43841
	35	0.95701	0.77206	0.61067
	42	0.53573	0.39498	0.41512

The area roughness of the surface of the coating was recorded based on its arithmetical mean height, S_a. The lowest surface roughness, 0.39498 µm was obtained when synthesised at 42 mA/cm^2 for 30 minutes. This agrees with Koay's findings that 30 minutes of deposition time produced samples with the least roughness (Koay et al., 2017, Hyie et al., 2016). Figure 1 shows that the lowest current density, 28 mA/cm^2 has modest area roughness that sits between 42 mA/cm^2 and 35 mA/cm^2.

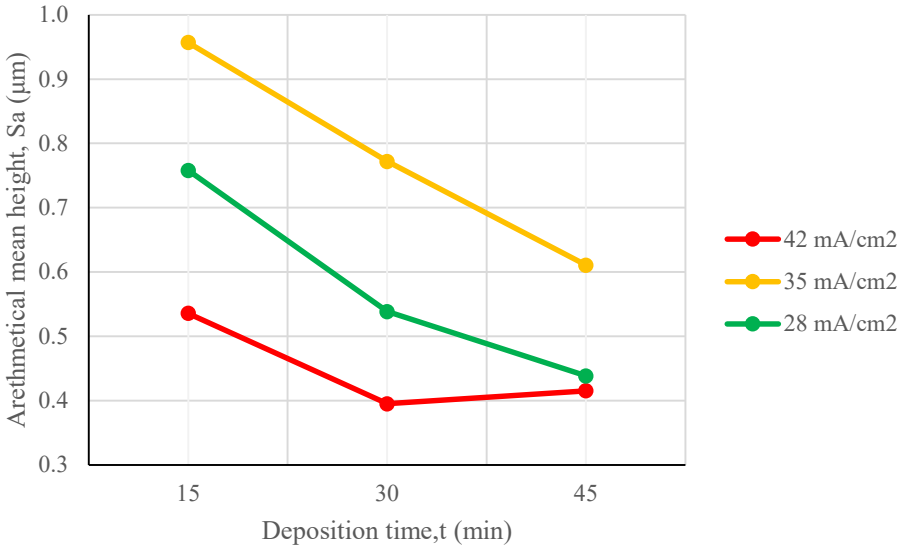

Figure 1: Effect of deposition time and current density on the surface roughness.

At 35 mA/cm^2, the deposition time of 15 minutes produces a thin film with relatively high surface roughness (S_a = 0.95701 µm). In contrast, the coatings deposited for 30 minutes were characterised by their smooth surface with a lower value of roughness than 15 minutes (S_a = 0.61067 µm). The thin film synthesised in 30 minutes exhibits the same dense and smooth surface morphology (S_a = 0.77206 µm).

Generally, the longer the deposition times, the lower the surface roughness. However, 45 minutes of deposition time at 42 mA/cm^2 produced an undesirable result as it had the roughest surface and the value increased instead. Similar behaviour in which an unusual trend occurs at 45 minutes of deposition time with the assumption that levelling ability is the lowest at this point (Hyie et al., 2016). Based on the optical observation shown in Figure 2, there are many craters scattered all over the surface. These craters are around 4 µm deep and can reach as low as 11 µm as shown in a scale of Figure 3. Besides, the longer deposition time contributed to a thicker coating with the formation of large voids and higher protrusions.

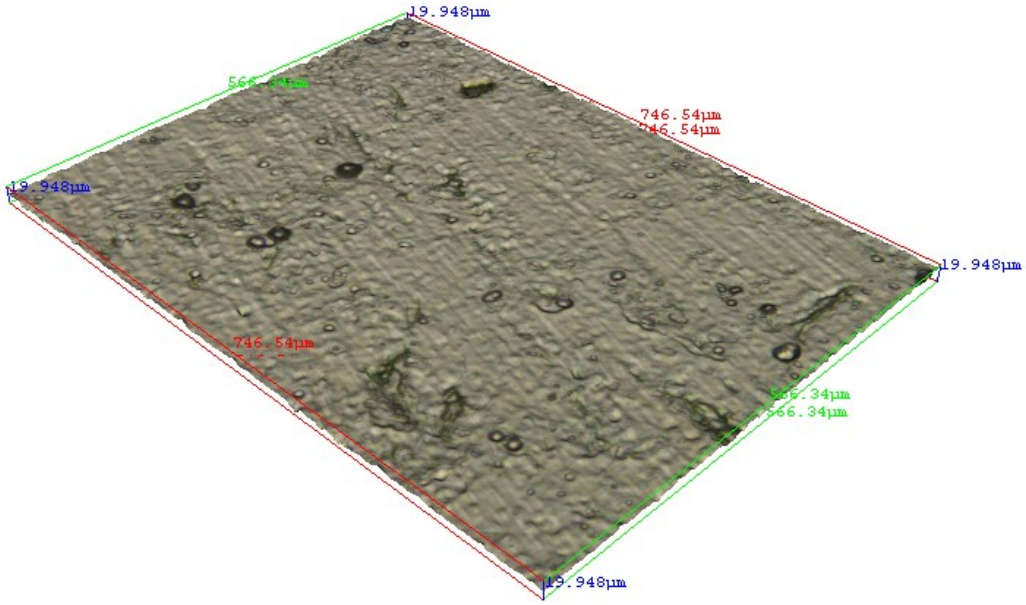

Figure 2: 3D images of coating synthesised at 42 ma/cm2 for 30 minutes in grayscale.

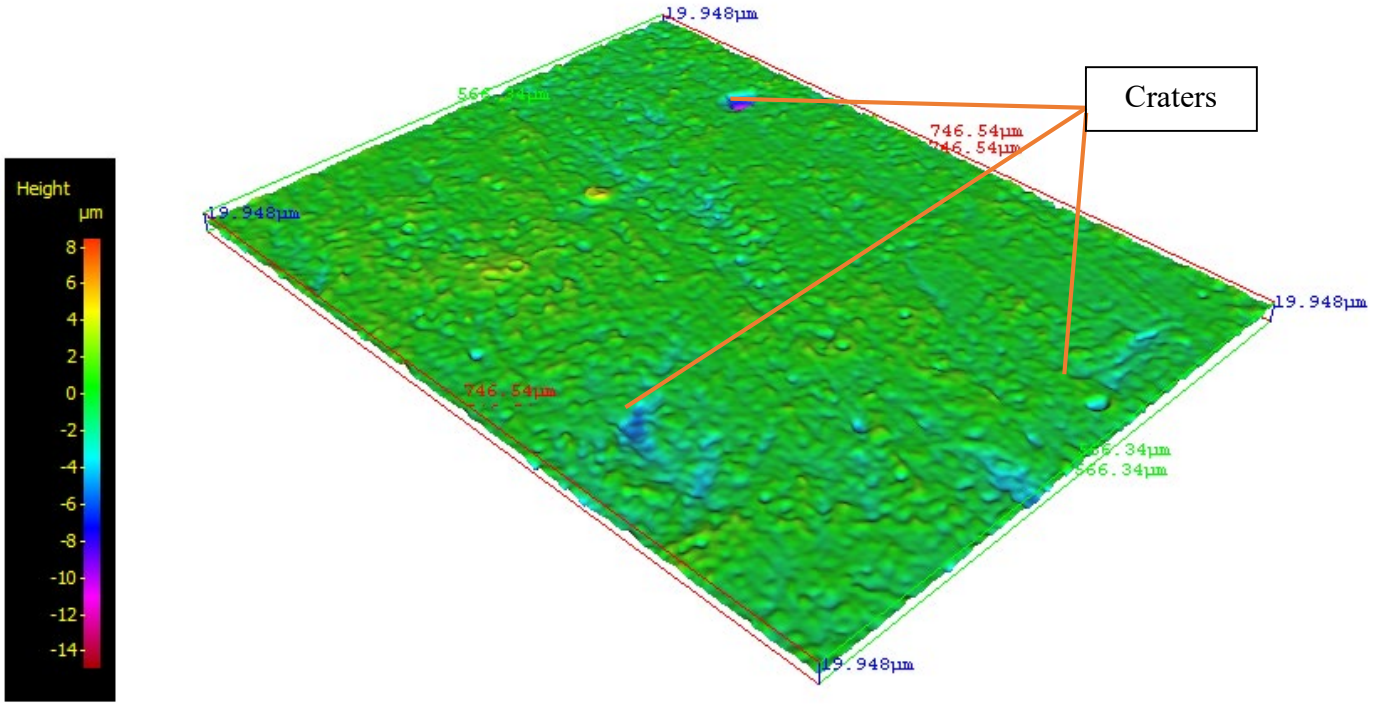

Figure 3: 3D images of coating synthesised at 42 ma/cm2 for 30 minutes in pseudo colour.

3.2 Microhardness

Figure 4 shows the microhardness of samples in Vicker's scale as a function of deposition time and current density. The hardness across all samples increased gradually with the increase of deposition time and current density. Coating synthesised at the highest deposition time and current density had the highest hardness value at 457.1 ± 27.1 HV. On the other hand, using parameters with the lowest deposition time and current density resulted in the lowest hardness value at 269.8 ± 32.6 HV.

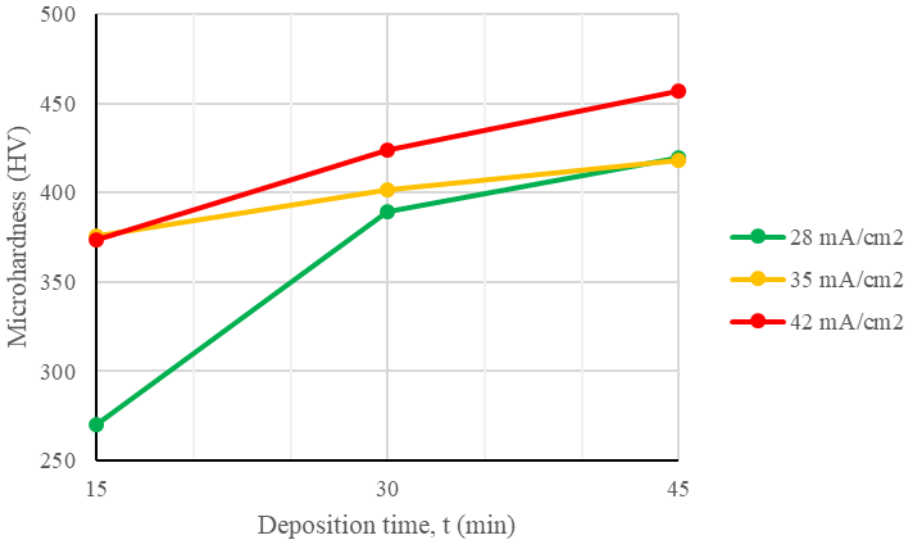

Figure 4: Microhardness of samples at different deposition times and current density.

Based on Figure 4 each line represents a current density. The higher the current density, the higher the hardness value. The current density line of 35 mA/cm^2 has the lowest gradient. It intercepts with lines 28 and 42 mA/cm^2 because sample T15-CD35 had higher and lower hardness than T15-CD42 and T45-CD28 respectively. At a deposition time of 15 minutes, the hardness difference between 35 and 42 mA/cm^2 is negligible at 0.43%. At 30 minutes, the hardness difference is 35 and 28 mA/cm^2 is 3.45%. This difference makes the current density of 35 mA/cm^2 a transition current density.

There is a high gradient in the current density line of 28 mA/cm^2 which is a significant increase in hardness between 15 and 30 minutes of deposition time. It is influenced by a porosity effect. A loose atoms arrangement introduces a weak link in the lattice structure that reduces the hardness of the coating. A previous study reported that voids and agglomerates reduce the hardness value of Co-Ni-Fe coating (Hyie et al., 2016). Figure 5 shows the presence of irregular grain structure and size which contribute to a low hardness value. Thus, the sample synthesised using a current density of 28 mA/cm^2 for 15 minutes has a sub-par hardness value compared to the rest of the sample.

A high hardness value is influenced by the uniformity of the coating which can be observed in Figure 6. A compact and complete crystal lattice have less agglomerate and voids (Resali, Koay, Berhan, & Mardziah, 2014). This condition improves the hardness Co-Ni-Fe nanocrystalline when it is synthesised using longer deposition time and higher current density. Some factors that contribute to the increase of the microhardness value between the sample are surface composition and size of the grain. Based on Figure 6, samples T45-CD42 have a smooth surface finish and fewer defects than T15-CD28. The surface was smoother because grain boundaries formed at the nanoscale instead of the microscale. These boundaries act as a deterrent to change or stop the dislocation movement (Koay et al., 2017). Thus, the coating hardens and plastic deformation in the material can be avoided.

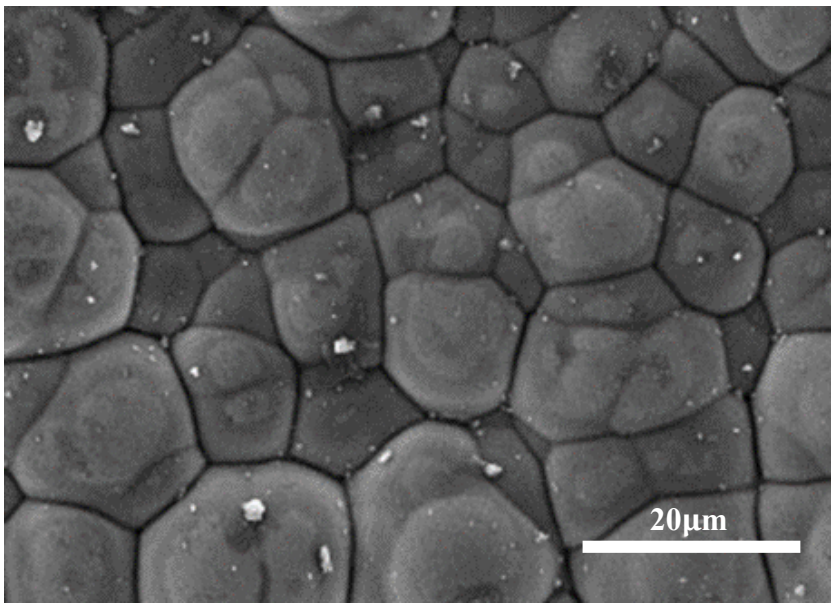

Figure 5: A micrograph of sample T15-CD28

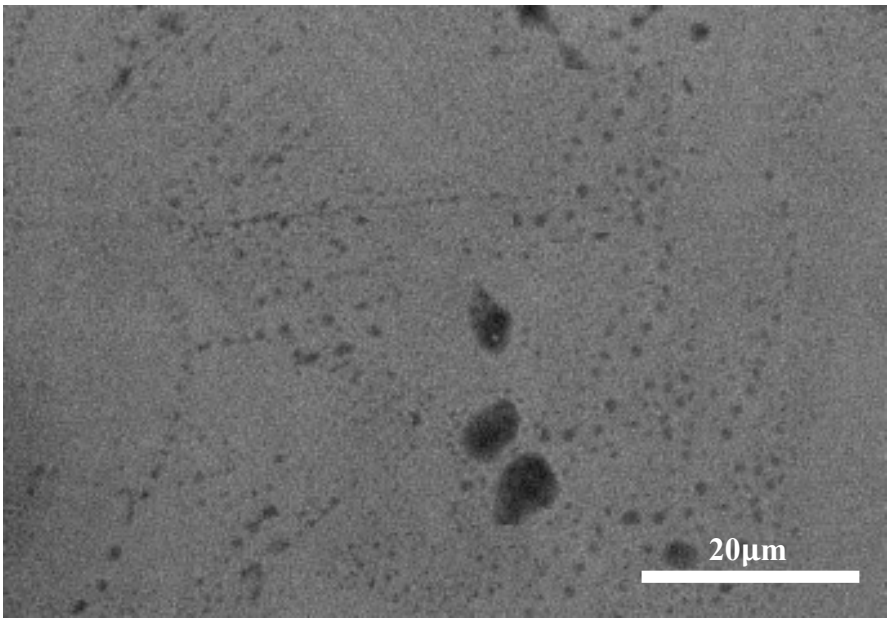

Figure 6: A micrograph of sample T45-CD42

The elemental content in the synthesised coating is a deciding factor of hardness value too. Iron (Fe) is known for its high brittle criteria which contribute to the increase of hardness of the coating (Hadian & Gabe, 1999). Figures 7 and 8 show the Co, Ni and Fe spectrum of the sample with the lowest and highest hardness value respectively. The Fe content in sample T15-CD28 was 8.7 wt% and sample T45-CD42 was 16.2 wt%. The increment in Fe content contributed to the 69.4 % increase in hardness value. Besides, Fe content also influenced the morphology. A higher Fe content resulted in a more uniform structure as shown in Figure 6. A similar trend was also reported where a higher Fe content produced a more compact surface topography (Saraç & Baykul, 2021).

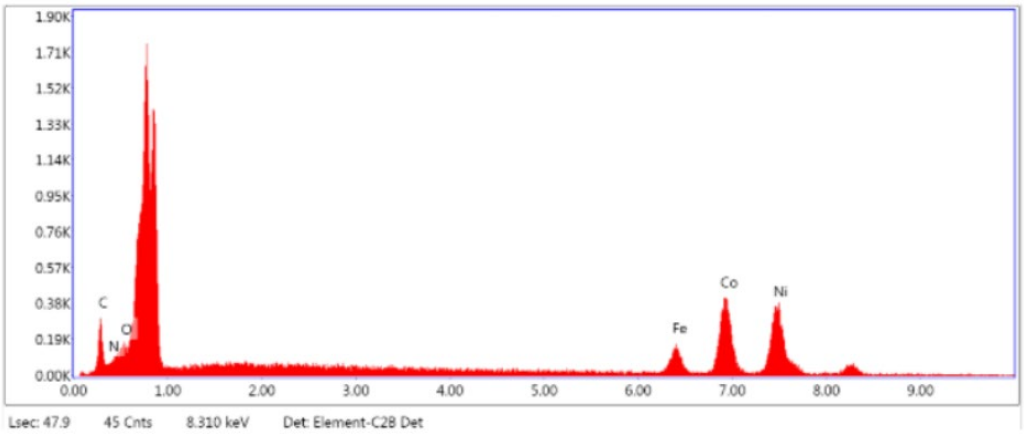

Figure 7: EDX spectrum of sample T15-CD28.

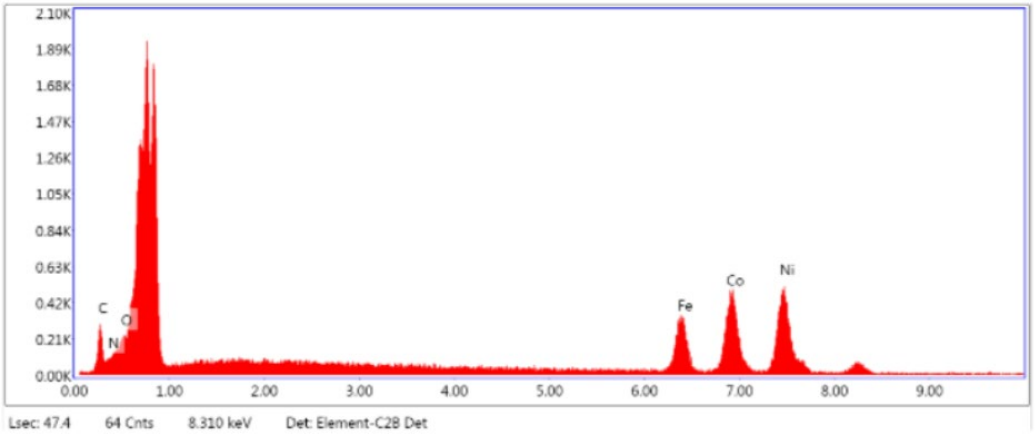

Figure 8: EDX spectrum of sample T45-CD42

4 Conclusion

There was a direct relationship between deposition time and surface roughness obtained. On the other hand, current density did not have a direct relationship with surface roughness. Generally, a lower surface roughness contributed to better corrosion resistance. When the current density was increased, the microhardness increased. The highest hardness value (HV) recorded was 285.3 HV whereas the lowest HV was 126.4 HV. EDX spectrums showed the content of cobalt, nickel and iron was lower at lower current density. SEM micrographs indicated that a homogenous coating of Co-Ni-Fe particles is achievable when deposited using a current density of 42 mA/cm² at 30 or 45 minutes only. A minimal presence of void or agglomerates proves that the Co-Ni-Fe coatings could improve microhardness and surface morphology. The current density of 35 mA/cm² gave the highest microhardness but its surface morphology was sub-par for corrosion resistance properties. On the other hand, the current density of 28 mA/cm² produced a decent surface morphology but its microhardness was among the lowest. In terms of application, these bolts will be used as fasteners for granite dust-filled glass fibre pipes in the future.

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6 Availability of Data and Material

Data can be made available by contacting the corresponding author.

7 References

Finšgar, M., & Jackson, J. (2014). Application of corrosion inhibitors for steels in acidic media for the oil and gas industry : A review. *Corrosion Science*, *86*, 17–41. https://doi.org/10.1016/j.corsci.2014.04.044.

Hadian, S. E., & Gabe, D. R. (1999). Residual stresses in electrodeposits of nickel and nickel–iron alloys. *Surface and Coatings Technology*, *122*(2–3), 118–135. https://doi.org/10.1016/S0257-8972(99)00328-X.

Hyie, K. M., Resali, N. A., & Abdullah, W. N. R. (2012). Study of Alloys Addition to the Electrodeposited Nanocrystalline Cobalt. *Advanced Materials Research*, *486*, 108–113. https://doi.org/10.4028/www.scientific.net/AMR.486.108.

Hyie, K. M., Zabri, M. Z., Nik Roseley, N. R., & Nik Mohd Masdek, N. R. (2016). Effect of deposition time on wear and corrosion performance of Co–Ni–Fe alloy coated mild steel. *Journal of Materials Research*, *31*(13), 1848–1856. https://doi.org/10.1557/jmr.2016.32

Koay, M. H., Tukiran, M. A. A., Mohd Halidi, S. N. A., Che Murad, M., Salleh, Z., & Yusoff, H. (2017). Corrosion investigation of Co–Ni–Fe-coated mild steel electrodeposited at different current densities and deposition times. *Industrial Lubrication and Tribology*, *69*(3), 393–398. https://doi.org/10.1108/ILT-09-2016-0215.

Koch, G. (2017). Cost of corrosion. In *Trends in Oil and Gas Corrosion Research and Technologies* (pp. 3–30). Elsevier. https://doi.org/10.1016/B978-0-08-101105-8.00001-2.

Lowenheim, F. A. (2014). *Modern Electroplating*. (M. Schlesinger & M. Paunovic, Eds.) (5th ed.). Hoboken: John Wiley & Sons, Inc. https://doi.org/10.1149/1.2425993.

Resali, N.A., Koay, M. H., Berhan, M. N., & Mardziah, C. M. (2014). Investigation of Heat Treated Electrodeposited CoNiFe on Microstructure and Hardness. In *Proceedings of the International Conference on Global Sustainability and Chemical Engineering* (pp. 227–233). https://doi.org/10.1007/978-981-287-505-1.

Resali, Nor Azrina, Hyie, K. M., Berhan, M. N., Salleh, Z., & Kasolang, S. (2013). Cobalt-nickel-iron Nanoparticles Coated on Stainless Steel Substrate. *Procedia Engineering*, *68*(12), 30–36. https://doi.org/10.1016/j.proeng.2013.12.143.

Saraç, U., & Baykul, M. C. (2021). Studying structural, magnetic and morphological features of electrochemically fabricated thin films of Co–Ni–Fe with different Fe compositions. *Thin Solid Films*, *736*, 138901. https://doi.org/10.1016/j.tsf.2021.138901.

Tabakovic, I., Gong, J., Riemer, S., & Kautzky, M. (2014). Influence of Surface Roughness and Current Efficiency on Composition Gradients of Thin NiFe Films Obtained by Electrodeposition. *Journal of the Electrochemical Society*, *162*(3), D102–D108. https://doi.org/10.1149/2.0351503jes.

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