Surface Properties and Characterisation of Cobalt-Nickel-Iron Alloy Coating on Mild Steel for Engineering Applications

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ABSTRACT: Corrosion prevention, enhancement of mechanical properties and aesthetic improvement are key motivators for the use of protective coatings in engineering applications. This study investigates the surface characteristics of electrodeposited Cobalt-Nickel-Iron (CoNiFe) nanoparticle coatings on mild steel substrates to assess cobalt alloy potential for improved performance and durability. Through the usage of electrodeposition method, coatings were applied at varying deposition times (15, 30, and 45 minutes), with electrolyte temperatures maintained at constant 50 ± 3 °C and current levels set at 1.0, 1.5, and 3.0 A respectively. Key properties evaluated include surface roughness, hardness, coating thickness, and breaking energy. Results showed that coating thickness ranged from 14.23 µm to 40.80 µm, with the thickest coating recorded at 45 minutes' deposition time. Hardness value also increased at longer deposition durations, with the highest achieved value of 503.12 HV at 45 minutes' deposition time. Surface roughness ranged from 1.4210 µm (30 minutes, 1.0 A) to 8.1085 µm (45 minutes, 3.0 A), indicating significant influence of deposition conditions. Additionally, the coating had a maximum breaking energy of 74 Joules at high-temperature quenching conditions (30 minutes). These findings suggest that CoNiFe coatings applied via controlled electrodeposition process can significantly improve the mechanical resilience and lifespan of mild steel, offering promising applications for advanced engineering environments.

KEYWORDS: Cobalt-Nickel-Iron, Deposition times, Surface roughness, Surface properties, Hardness

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I. INTRODUCTION

Coating is a type of surface covering that is applied to a substrate. Coating is critical for machine components because of its capability to enhance parts life and protect it against corrosion (Hyie *et al.*, 2022). Coatings are widely known for their importance in corrosion prevention because they prevent steel surface from being directly exposed to air or the environment (Zabri *et al.*, 2022). Electroplating, also known as electrodeposition, is a simple method that involves applying a thin layer of nanoparticles to a base metal in order to improve the material properties (Md. Nor *et al.*, 2019). Electrodeposition is well recognised for its fast-coating production (Zankowski, 2019). This approach offers numerous advantages, including a low cost, ease of control over the nanocoating production process and high deposition rate (Bauer *et al.*, 2023). The desired coating characteristic and performance are achieved by modifying the processing parameters: electrolyte composition, current density, temperature, pH, and deposition time (Md.Nor *et al.*, 2021; Gao *et al.*, 2015).

There are many metals that are compatible with mild steel and can be easily shaped into coating layers. Many industrial and technological applications favour ferromagnetic alloys made of Co, Ni, and Fe because they have good magnetic, structural, mechanical, and morphological qualities (Saraç et al, 2021). Nickel-cobalt (Ni-Co) is a suitable nickel-based alloy for coatings. In comparison to other nickel-based alloy coatings, the dense Ni-Co alloy solid solution demonstrates greater structural stability, corrosion resistance, and mechanical properties when Ni and Co are combined (Tan *et al.*, 2022). It is also simple to make Ni- Fe binary composite coatings using the electrodeposition method. This approach has some advantages, including better control over alloy composition, coating thickness, and shape (Solmaz and Kardaş, 2009).

A previous study of electrodeposited CoNiFe nanoparticles revealed that the process parameters have a substantial impact and advantages on the material's microstructure and properties (Resali *et al.*, 2013). Electrolyte material, temperature, pH level, deposition duration, and current density are all easily controllable experimental factors (Zabri *et al.*, 2022; Solmaz and Karahan, 2021). The pH of the bath has a major impact on both the operation of the bath and the composition of the coating. For example, the Nickel content of the deposit increases as the alkalinity of the solutions increases (Loto, 2016). Higher pH values produce deposits with less phosphorus, while lower

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pH ranges produce deposits with a lot of phosphorus (Loto, 2016).

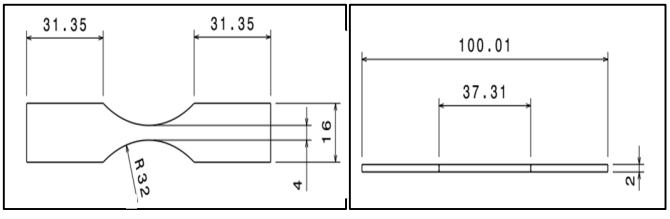
Recent research has also demonstrated that altering current density can improve the hardness and corrosion resistance of CoNiFe electrodeposited coatings, making them a viable alternative to standard chromium coatings (Wang *et al*., 2024). Wang *et al.* (2024) reported that adding an additive such as Saccharine can contribute to a flat and brilliant surface, increase anti-corrosion, and minimise internal stress in the coating. Haizad *et al.* (2023) further highlighted that corrosion rates vary with the environment through their finding in which CoNiFe coatings showed lower corrosion rates in freshwater compared to seawater. The CoNiFe coatings heat-treated at 400°C exhibited improved hardness and corrosion resistance, while those treated at 1000°C showed a drastic reduction in tensile strength (Haizad *et al.*, 2023). Thus, it is assumed that with the combination of CoNiFe alloy, it will give the potential to replace other market materials as an alternative coating with better surface attributes comparable to chromium coating.

However, most of the previous work focused on implementing CoNiFe nanoparticles coating on substrates other than dog bone and notch block samples which will be the focus area in this work. Therefore, this study examines surface characteristics of the electrodeposited CoNiFe nanoparticles coated mild steel samples utilising a different deposition duration at a constant pH and temperature. Concurrently, the characteristics of several identical coated duration samples were examined further to determine the effect of varying current at constant deposition time. The impact of the introduction to the post-coating procedure on the energy required to break the selected coating samples is also being investigated throughout this study.

II. METHODOLOGY

A. Sample preparation

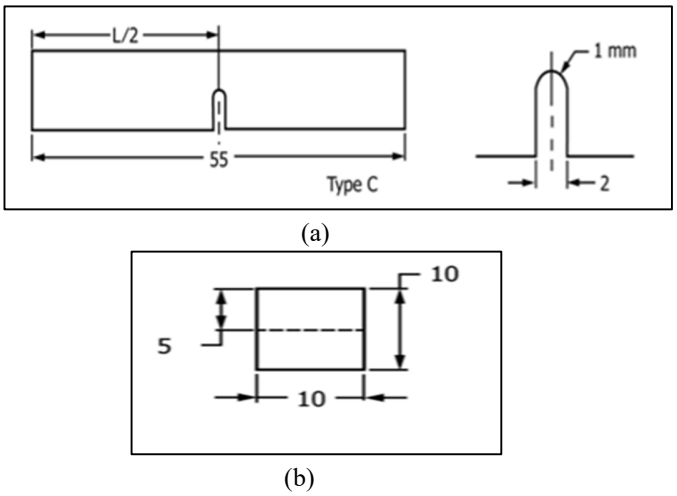
Material used for the experiment was a mild steel substrate. Mild steel substrate was cut into two types of dimensions (i) 100 mm x 20 mm x 3 mm and later cut accordingly into dog bone shape following ASTM E466 dimension using water jet cutter (ASTM, 2021). (ii) 55 mm x 10 mm x 10 mm block with notch 5 mm x 2 mm following ASTM E23 (ASTM, 2024).



(a) (b)

Figure 1: Dimension of dog bone substrate in unit mm (a) top view; and (b) front view.

CoNiFe nanoparticles coating was deposited by electrodeposition by placing mild steel substrate at cathode, while a platinum plate mesh was used as the anode. Prior to plating, the mild steel substrate was first grind using 120, 220 and 320 grit flap disks, followed by cleaning with a clean microfiber cloth soaked in ethanol. The substrate was then rinsed with distilled water, at room temperature. Figures 1 (a-b) and 2(a-b) show the detail dimension of dog bone and charpy impact test substrate used for the study.



(a)

(b)

Figure 2: Dimension of charpy impact test substrate in unit mm (a) top view; and (b) side view.

B. Electrodeposition process

The CoNiFe nanoparticles coatings were created by electroplating CoNiFe nanoparticles coatings from a sulphate bath. The sulphate bath was made by combining sulphate-based powders such as Cobalt sulphate ($CoSO_4$), Nickel sulphate ($NiSO_4$), Iron sulphate ($FeSO_4$), and Ascorbic acid ($C_6H_8O_6$). Addition of remaining two powders inside the bath such as Saccharine ($C_7H_5NO_3S$) and Boric acid (H_3BO_3) act as grains refinement agent and pH buffer respectively (Zabri, M. Z. *et al.*, 2023). The detail of chemicals utilised to prepare an electrolyte for the coating process is presented in Table 1.

Table 1: Chemicals utilized to prepare electrolytes.

Compound	No. of Moles
Cobalt Sulphate ($CoSO_4$)	0.050
Nickel Sulphate ($NiSO_4$)	0.133
Iron (II) Sulphate ($FeSO_4$)	0.020
Boric Acid (H_3BO_3)	0.267
Ascorbic Acid ($C_6H_8O_6$)	0.067
Saccharine ($C_7H_5NO_3S$)	0.007

Throughout the electrodeposition process, mild steel substrates and platinum mesh plate were connected to the cathode and anode, respectively. Constant and fixed variables were continuously monitored and regulated during the electrodeposition process such as pH, temperature and current setting. The pH of the electrodeposition bath was manipulated by appropriate additions of potassium hydroxide (KOH)

solution. Figure 3 depicts a schematic diagram of the electrodeposition process setup.

The CoNiFe nanoparticles deposition times were set to 15, 30 and 45 minutes, and the current was kept varied from 1.0 A, 1.5 A and 3.0 A. The chemical components were mixed with distilled water to form 1 liter of electrolyte solution and heated with a hotplate to 50°C – 53°C. Throughout the electroplating process, it is vital to keep an eye on the electrolyte solution's temperature.

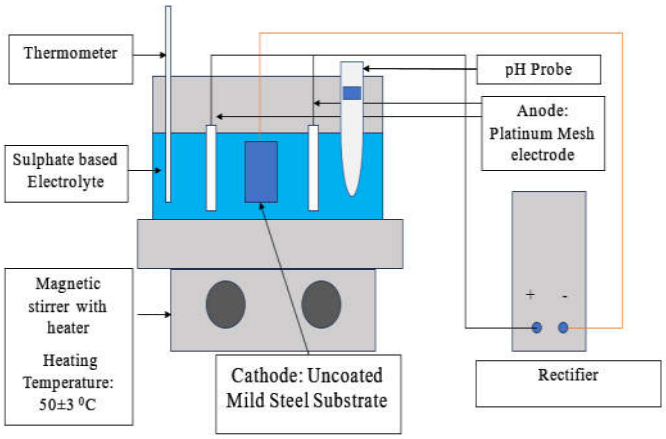


Figure 3: Schematic diagram of electrodeposition process setup.

The solution's composition may change as a result of overheating, and the coating material formed on the substrate may decrease in quality. Table 2 summarises the parameters involved throughout electrodeposition process.

Table 2: Parameters involved for electrodeposition process.

Parameters	Control Setting
Arrangement of Electrode	Anode: Platinum wire mesh Cathode: Mild steel substate
Temperature	50°C -53°C
Electrolyte pH	3 (fixed)
Current used	1.0, 1.5 and 3.0 A
Deposition time	15, 30, and 45 minutes

C. Testing preparation

The surface morphology of the CoNiFe nanoparticles coatings were observed via field emission scanning electron microscope (FESEM) (Thermofisher) at an accelerating voltage of 10 kV and targeting magnification of 200,000 times magnification. Vickers hardness was evaluated using a Mitutoyo MVK-H1 machine, while surface roughness was determined using a Surf test SV600 machine. Vickers hardness data is obtained by indenting the specimen for 5 seconds with a diamond indenter loaded with 1 kg.

Surface roughness value recorded by applying a straight-line contact using diamond probe on the substrates surface. Five separate measurements were made for both Vickers hardness and surface roughness data at various spots on substrate surfaces, and the average hardness and surface roughness value were determined. The surface roughness were measured by dragging the needle (or stylus) along the surface for roughly 5 mm. Three distinct readings were collected at different positions on a sample's surface for these tests, and the average value of hardness was determined. Based on the surface roughness value, the potential sample were chosen and tested further using charpy test. From the charpy test, the energy required to break the samples were determined.

Physical observation of the samples during exposure to salt water are achievable by using salt fog test. 318 g of sodium chloride (NaCl) with 6 litres of distilled water were mixed following ASTM B117 standard (ASTM, 2019). The operating temperature was held constant at 35°C ± 3°C, while the pH value was regulated between 6.5 and 7.2. All samples were left for 48 hours in the salt spray (fog) test chamber before been removed. The sample was removed from the salt fog chamber, washed under distilled water at a temperature no higher than 38°C (100°F) to remove any salt deposits from their surface, and then dried immediately.

Specimens prepared in accordance with ASTM E23 were utilised for the Charpy Impact test (ASTM, 2024). The notch on the specimen acts as a point of stress concentration, which is essential for accurately measuring the energy value. The results of the Charpy impact test are obtained by releasing a heavy pendulum at a constant swing angle of 135° using a Charpy testing machine. Samples fabricated according to ASTM E23 were coated with CoNiFe nanoparticles prior to being positioned horizontally on supports, with the notch oriented away from the direction of impact (ASTM, 2024). Upon striking the specimen at high velocity, the pendulum causes it to fracture at the notch. The machine quantifies the energy absorbed by the specimen during this fracture, determined by the difference between the pendulum's initial and final heights. This absorbed energy, generally expressed in Joules, is recorded as the material's impact toughness, providing valuable information regarding the material's capacity to endure sudden impact forces.

III. RESULTS AND DISCUSSION

A. Field Emission Scanning Electron Microscope

The field emission scanning electron microscope (FESEM) images were used to determine the morphology of the CoNiFe nanoparticles coating. Figures 4(a), (b) and (c) display FESEM images of a CoNiFe alloy coating at various deposition times, including 15, 30, and 45 minutes, utilising a 200,000X magnification resolution at 10 kV. The 30 minutes coated sample created a full and uniform structure, as shown from the microstructures. This is owing to the ordered arrangement of atoms during adequate deposition time (Hyie *et al*., 2016). The existence of gaps or porous areas as indicated by black region on the substrate surface of the 15 minutes coated sample suggests that the surface was not completely covered with CoNiFe nanoparticles. Hyie *et al.* (2016) discovered the similar finding that void existed on sample coated at lower deposition time due to the insufficient deposition time to cover the surface of sample.

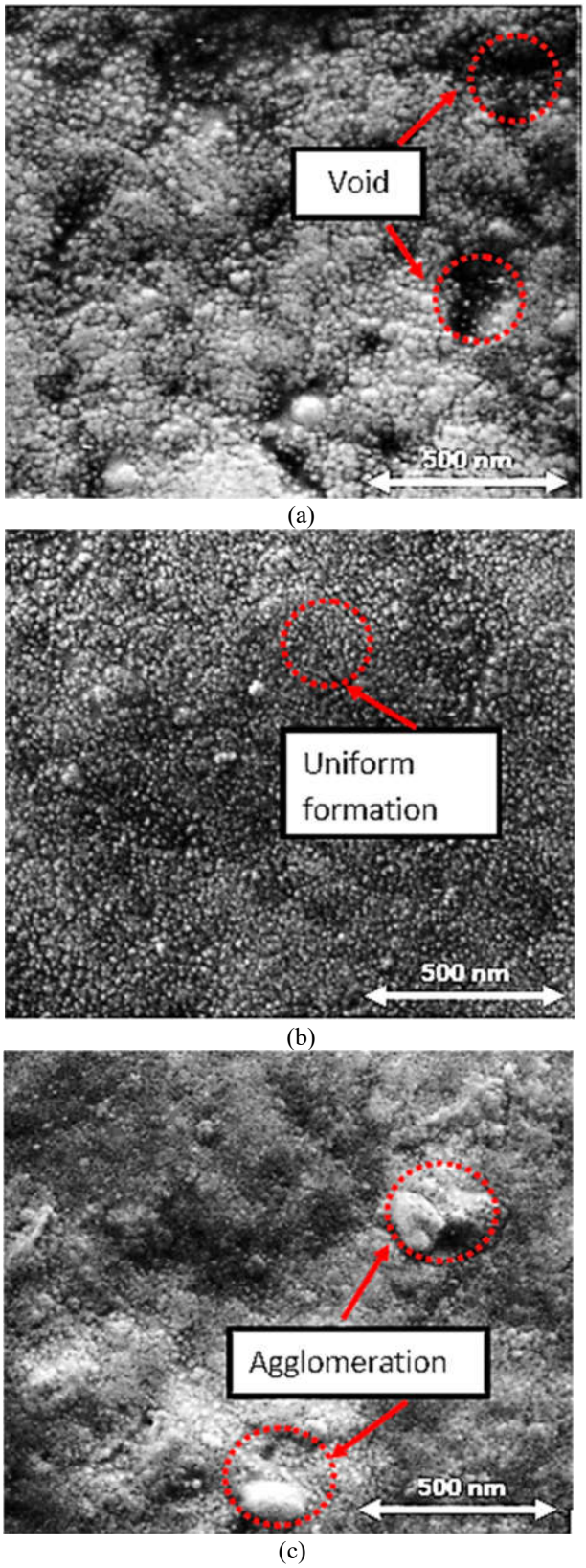


Figure 4: FESEM Images of CoNiFe nanoparticles coating at (a) 15 minutes; (b) 30 minutes; and (c) 45 minutes deposition times.

It is worth knowing that the particles must have enough time to spread evenly and rearrange themselves within the microstructure before the ions from the electrolyte solution can be reduced and the coating can be deposited. If the deposition period is shorter, it would not have enough time to create a full coating dissemination, leaving some substrate areas vacant and less coated. Globular-like structure with varying dimensions and nanoparticles stacking are clearly observed on 45 minutes coated sample. The existence of irregular shape in the microstructure, in general, has cultivated an interface between grain boundaries. As the deposition times increases, the particles become larger and more likely to mix and form stacking formation and agglomeration (Hyie *et al.,* 2016). Resali *et al.,* (2013) concluded that Cobalt alloy nanoparticles prepared at higher deposition times were larger and intended to combine and coalesce with each other to form agglomerates

B. Coating Thickness

The thickness varies across three deposition times listed in Table 3. For 15 minutes deposition times, coating had thickness of 14.23 µm whereas the coating with 25.23 µm and 40.80 µm thickness was synthesised for 30 minutes and 45 minutes deposition times, respectively. Theoretically, the values are acceptable as more CoNiFe nanoparticles coatings could be deposited on the mild steel substrate due to a longer deposition period, which increased the coating's thickness (Resali *et al.,* 2013). Murmu *et al.,* (2022) experienced increment in coating thickness as deposition time increased. The surge of thickness change is also observed by Nor *et al.,* (2021) at different deposition times.

Table 3: Recorded thickness of the samples

Type of Sample	Average Coating Thickness (µm)
15 min - 1.5 A (pH 3)	14.23
30 min - 1.5 A (pH 3)	25.23
45 min - 1.5 A (pH 3)	40.80

C. Salt Fog Test

The results of the salt fog spray in Table 4 clearly show that the uncoated sample has the highest degree of corrosion compared to the CoNiFe nanoparticles coated samples. The 15 minutes coated sample shows a large light brown band of rust, whereas the 30 minutes sample has numerous small rust spots, and the 45 minutes sample shows the least damage of rust. The appearance of brown rust in a 45 minutes sample indicates that corrosion is still in its early stage.

According to Nor *et al.,* (2018), mild steel bolt that is coated for 45 minutes has the superior corrosion resistance when compared to samples that are coated for 15 minutes and 30 minutes. After 48 hours of salt spray testing, the brown color is barely visible on the surface, and the CoNiFe coating is still present. Theoretically, the longer the coating deposition time, the greater the coating's resistance. This is due to the fact that a longer deposition time results in a thicker coating on the sample. The thicker coating will form a longer diffusion

barrier and prevent corrosive substances from reacting with the substrate, resulting in rust. Murmu *et al.* discover that the rate of corrosion correlate with surface roughness and higher recorded surface roughness correspond to a lower corrosion rate (Murmu *et al*., 2022). To summarise, the findings of this investigation were consistent with theory and past related research.

Table 4: Physical observation on the samples after being exposed under 48 Hours Salt fog test.

Type of sample	Visual rust condition
Raw Mild Steel	
Deposition times: 15 Minutes	
Deposition times: 30 Minutes	
Deposition times: 45 Minutes	

D. Surface Roughness Test

For coating with fixed 1.5 A applied current as in Figure 5, the 30 minutes coating sample had the lowest surface roughness of 1.4210 µm, whereas the 45 minutes coating sample had the greatest surface roughness of 4.0653 µm. The decreased in surface roughness at 30 minutes deposition times might be attributed to homogeneous particle formation with appropriate particle size and dense population as a result of the increase in nucleation density (Lee and So, 2000). The surface roughness value may vary based on the FESEM morphologies because of the void and agglomeration that were present in the 15 and 45 minutes coating samples, respectively. Despite the fact that deposition times increased both hardness and coating thickness, the resulting surface roughness did not follow the same pattern (Kato *et al*., 2014).

Figure 6(a) depicts the average surface roughness of CoNiFe nanoparticles coating at 15 minutes deposition times (different current setting at I: 1.0 A and 1.5 A). For 15 minutes deposition times with varied current case, higher applied current of 1.5 A reduced down the surface roughness from 5.8495 µm to 3.5559 µm, the decrease in surface roughness might be attributed to reduction of void which presence on 15 minutes deposition times with 1.0 A current into more homogeneous particle formation with appropriate particle size and dense population as the current increased to 1.5 A. Koay *et al.,* (2017) discovered that coating process required a longer time if the current density is set lower. If the coating duration was insufficient, voids in the coating created an uneven surface, resulting in a coarse surface coating. As the deposition duration and current density increased, the sample coating improved and fully formed, resulting in lower surface roughness due to fewer voids.

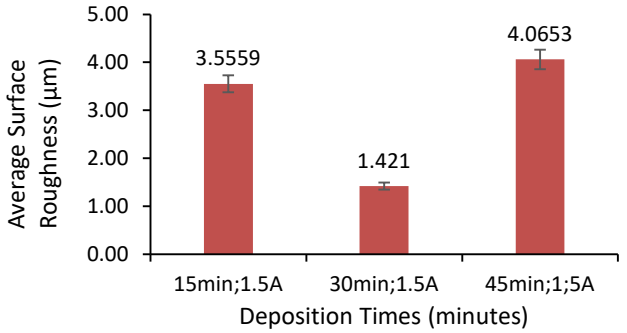
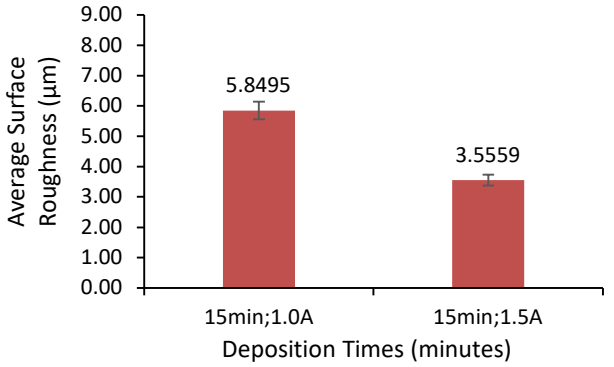
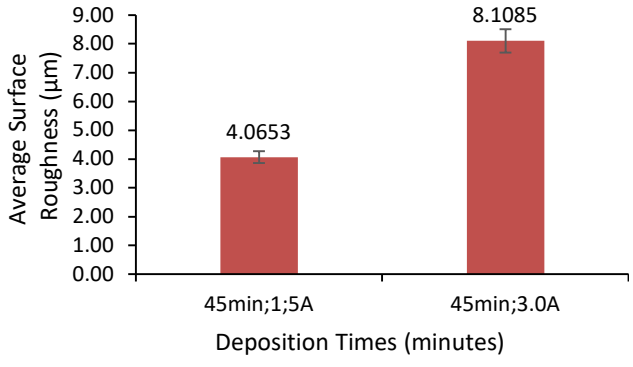


Figure 5: Average surface roughness of CoNiFe nanoparticles coating at 15, 30 and 45 minutes deposition times (similar current setting at @ I:1.5 A).



(a)



(b)

Figure 6: Average surface roughness of CoNiFe nanoparticles coating at (a) 15 minutes deposition times (different current setting at I: 1.0 A and 1.5 A); and (b) 45 minutes deposition times (different current setting at I: 1.5 A and 3.0 A).

Application of high current application at more higher deposition times as shown in Figure 6(b), on the other hand, aided to rapid substrate coating coverage which may lead to the problem involving agglomeration or particle stacking onto

each other (Chinnasamy *et al.*, 2001). It was noted that the agglomerated particle is one of the reasons for high roughness of deposits. These particles were combined and coalesced with each other to form agglomerates and bigger particles which in this case might cause sudden increased in surface roughness value. Thus, controlling the parameters of current density and deposition time is critical in order to produce a homogeneous and dense microstructure.

E. Vickers Hardness Test

According to a study by Masdek *et al.,* (2021), the grain size and iron content are responsible for the coating's microhardness. When the graph in Figure 7 was compared to each sample, the particles at 45 minutes of deposition durations were larger and able to form particle boundaries. These particle boundaries served as barriers to prevent motion from becoming dislocated and to regulate motion direction besides formed the harder microstructure layer (Hyie *et al.*, 2018). As the thickness and mass of the CoNiFe nanoparticles coated mild steel sample are more than those of the uncoated mild steel sample, the hardness of the coated mild steel will be

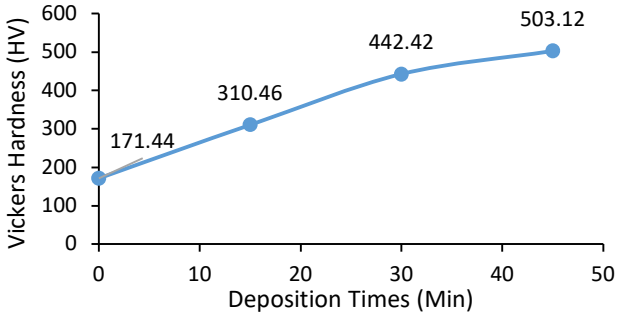


Figure 7: Vickers hardness against 15, 30 and 45 minutes deposition times with constant current.

greater. Hyie *et al.* (2016) discovered a similar tendency in which 45 minutes of deposition times showed increased hardness compared to 30 minutes and 15 minutes of deposition times. The alloy coating improved the hardness and consequently the surface performance of the mild steel (Masdek *et al.*, 2021). It can be concluded that the microhardness value increases as the deposition times increases.

It is also worth to know that as current increases, so does the hardness. As comparison in Figures 8 (a) and (b), hardness value for 15 minutes coated mild steel substrate with 1.5 A is higher than the similar samples coated with 1.0 A, which recorded 310.46 HV and 265.84 HV, respectively. Similar patterns were also seen on the 45 minutes coated mild steel substrate coated with 1.5A and 3.0 A which recorded 526.44 HV and 503.12 HV, respectively. This result is believed to be due to the lower tension associated with electrodeposited films (Nor *et al.*, 2021).

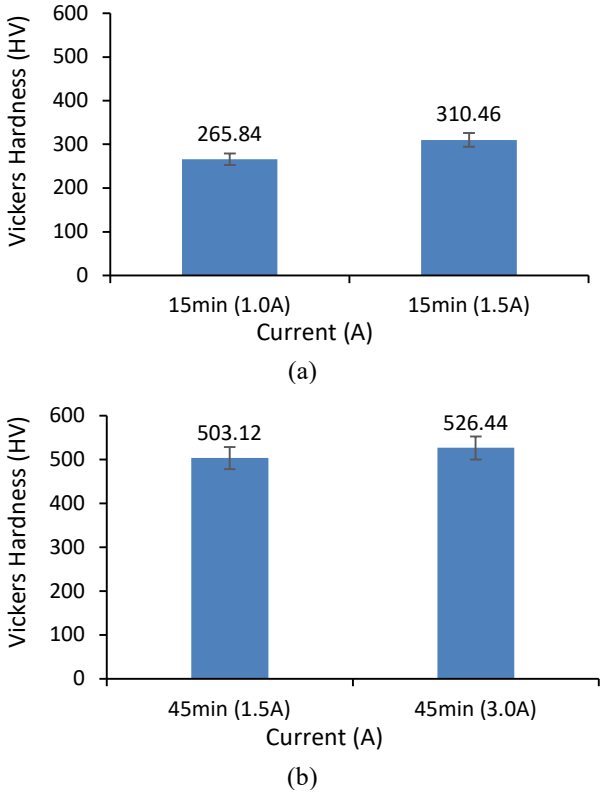


Figure 8: (a)Vickers hardness against constant 15 minutes deposition times with manipulated current setting (I: 1.0A and 1.5A); and (b) Vickers hardness against constant 45 minutes deposition times with manipulated current setting (I: 1.5A and 3.0A).

F. *Net Coating Mass Measurement*

As illustrated in Figure 9, coating applied with similar current setting; I:1.5 A showing the lowest coating mass recorded by 15 minutes deposition times (0.413 g). The highest coating mass recorded was coming from 45 minutes deposition times (1.144 g). It can be observed that ore CoNiFe nanoparticles coatings could be deposited on the mild steel substrate due to a longer deposition period, which increased the net coating's mass. Table 3 and Figure 9 show a direct correlation between coating mass and thickness. As the coating mass increased, so did the coating thickness.

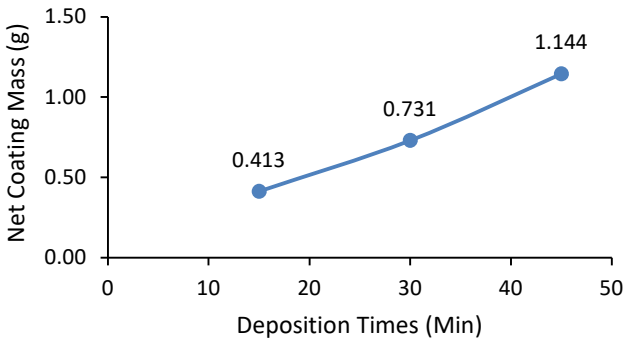


Figure 9: Net coating mass for 15, 30 and 45 minutes deposition times.

Figures 10(a) and (b) show the net coating mass for 15 minutes deposition times (different current setting; I:1.0 A and 1.5 A) and net coating mass for 45 minutes deposition times (different current setting; I:1.5 A and 3.0 A), respectively. In order to determine the effect of manipulating current setting towards coating mass trend, two identical samples with each 15 and 45 minutes deposition times respectively were made using two different current settings. For samples with 15 minutes deposition time, two types of current setting were applied (I:1.0 A and 1.5 A), while current setting of I:1.5 A and 3.0 A were used for 45 minutes deposition time samples. It can be clearly seen that the trend of net mass increased with the application of higher current.

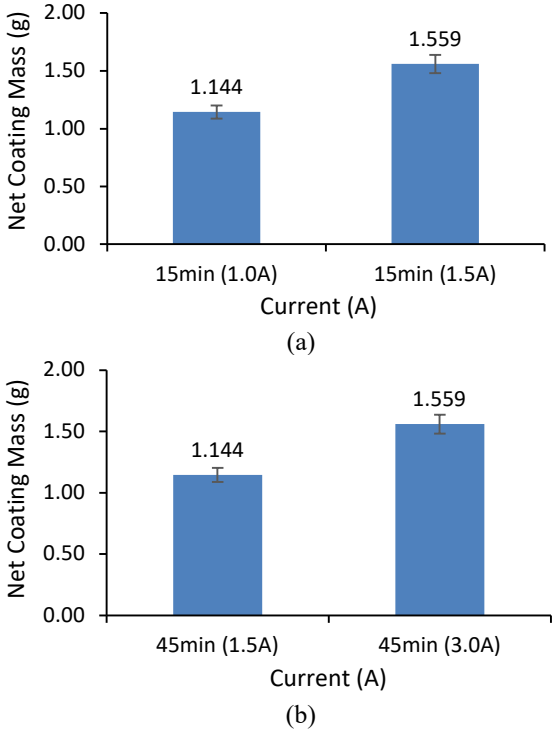


Figure 10: (a) Net coating mass for 15 minutes deposition times (different current setting; I:1.0A and 1.5A); and (b) Net coating mass for 45 minutes deposition times (different current setting; I:1.5A and 3.0A).

G. Charpy Test

Figure 11 depicts the impact energy recorded for raw mild steel and mild steel with deposition durations of 30 minutes (different post-coating procedure). The findings show that parameters selection and alteration, such as heat treatment techniques (quenching and annealing), increase the amount of energy required to break the samples. 30 minutes coating samples with heat treatment and quenching recorded the highest required energy of 74 Joules followed by 30 minutes coating samples with heat treatment and annealing, at 61 Joules. Application of annealing and quenching processes can cause difference in segregation, redistribution, and diffusion of elements within the coating. The rapid cooling rate of quenching can lead to the formation of harder surfaces besides reducing surface distortion and cracking (Lee *et al.*, 2022). This is typically attributed to the preservation of a finer microstructure. The CoNiFe nanoparticles coating in overall increased the amount of enery required to break the samples compared to the uncoated mild steel, which recorded lowest value of 49 Joules. The quenching media that was used is water for this study. Ogedengbe *et al.* (2022) discovered that the type of quenching media used during heat treatment could alter (optimise or reduce) the properties of the steel being heat treated. The usage of water, oil, brine and Potash Alum could obtain different result based on brittleness, and surface roughness. It is possible to evaluate a material's mechanical properties and resistance to impact loads using the specific impact energy values that were collected during the testing. These values show a material's capacity to absorb energy during impact.

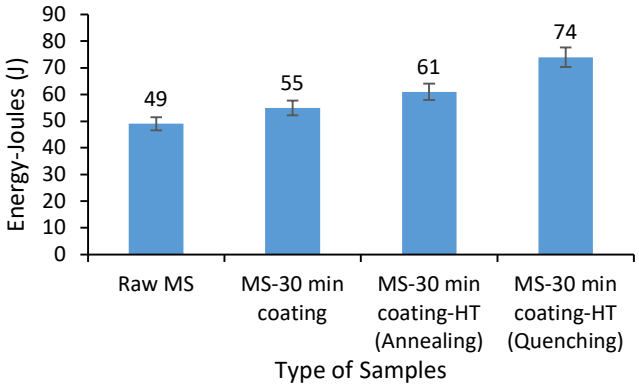


Figure 11: Energy recorded for Raw Mild Steel and Mild steel with 30 minutes deposition times (different cooling technique).

IV. CONCLUSION

This study investigated the effects of deposition time on the surface characteristics and performance of CoNiFe nanoparticle coatings on mild steel. The CoNiFe coating deposited at 30 minutes exhibited the lowest surface roughness and a homogenous, fine structure, as observed in FESEM images, indicating a uniform distribution of nanoparticles. In contrast, the coating prepared at 45 minutes achieved the highest microhardness and surface roughness values, as well as the largest net coating mass, suggesting a stronger yet rougher surface structure. The study also revealed that coating mass increased with longer deposition times and higher applied currents, with noticeable increments between current levels of 1.0 A, 1.5 A, and 3.0 A. The energy absorption test highlighted that the 30-minute coated sample, subjected to heat treatment followed by quenching, required the highest energy to fracture, indicating enhanced toughness compared to other samples. Both the 30-minute coating without heat treatment and with low-temperature cooling (annealing) showed improved energy absorption relative to the untreated mild steel substrate. These findings suggest that CoNiFe coatings deposited at 30 minutes provide a balanced enhancement of hardness, uniformity, and toughness, making them promising candidates for corrosion resistance and improved mechanical performance in engineering applications.

AUTHOR CONTRIBUTIONS

M.Z. Zabri: Conceptualization, Methodology, Validation, Writing – original draft. **N.R.N. Masdek:** Supervision, Writing – original draft. **C.M Mardziah and N.M Ahmad:** Writing – review & editing.

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