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# The Effect of Cobalt Alloy Nanocrystalline Coating on Tensile Properties and Surface Performance of Mild Steel

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ABSTRACT

*Coatings are frequently used to enhance the mechanical and physical characteristics of mild steel by protecting mild steel surfaces against corrosion. Corrosion might attack surface without proper coating method which may lead to structural failures and increase maintenance cost. The main objective of this study is to investigate the effect of CoNiFe nanocrystalline coating on surface roughness, hardness, and tensile performance of mild steel substrate. Different parameters were introduced, such as current, pH, deposition times and heat treatment to analyse surface roughness, hardness, and tensile strength of CoNiFe nanocrystalline coating. The lowest surface roughness of 2.14 µm was recorded by CoNiFe nanocrystalline coating with 30 minutes deposition time at pH 3, I: 3 A and heat treated, while the highest recorded surface roughness of 4.233 µm was detected on uncoated mild steel. The highest improvement of microhardness was observed on CoNiFe nanocrystalline coating with 45 minutes deposition time at pH 3, I: 1.5 A and heat treated (393.6 Hv) as compared to the uncoated mild steel (171.44 Hv). Tensile performance of CoNiFe nanocrystalline coating with 45 minutes deposition time at pH 3, I: 1.5 A and heat treated was the highest with yield stress and ultimate tensile stress of 472.35 MPa and 559.11 MPa, respectively. The lowest tensile performance was recorded by uncoated mild steel with yield stress and ultimate tensile stress of 149.40 MPa and 186.78 MPa, respectively.*

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*CoNiFe nanocrystalline coating has considerably improved the mild steel surface roughness, hardness and tensile strength which indicate superior mechanical properties for future engineering application.*

**Keywords:** *CoNiFe; Deposition Time; Tensile; Surface Properties; Hardness*

## Introduction

Surface coatings have been applied in several engineering applications to cordon and increases the lifespan of components exposed to corrosive environment [1]. In order to build, operate and protect electronics, machinery, construction and structural systems in harsh environments, nanoparticle alloy coatings with excellent mechanical and corrosion resistance are needed [2]. According to statistics, the annual maintenance cost for corrosion alone is estimated to cost millions of dollars, making it necessary to provide assistance and preventive measures to mitigate the consequences of erosion [3].

According to a study, uncoated metal surface cannot match the corrosion resistance and polarization resistance of nanocomposite coatings surface [4]. Therefore, adding a layer would significantly improve the strength and wear of mild steel. Electrodeposition technique is the best and most straightforward way of embedding nanocrystalline materials coating onto metal surfaces. In electrodeposition, an electric current is passed through a chemical solution to create an ionic solution, which allows the counter electrode's to be transferred to the working electrode's surface and form a coating. Electrodeposited coatings enhance the life cycle of the components used for various applications, besides being cost-effective, faster speed of deposition rate, and ease of control over the nanocoating fabrication process [5]. Popular electrodeposition materials such as Ni-P, Ni-W, Ni-P-W, Ag/Pd, Cu/Ag, Co/Ni, Co/Ag, and Zn-Ni are among the metals that have been extensively explored [6]-[8]. Co, Ni and Fe nanoparticles trio combination is chosen due to the existing excellent performance of each individual nanoparticles. Variable processing parameters such as electrolyte pH, temperature, deposition time, current density, and electrolyte composition govern the coatings' characteristics and performance during the electrodeposition process [9].

CoNiFe is being used for several applications. Yang et al. [10] in his research work on both air thermal stability and solar selectivity of CoNiFe oxide coating fabricated by facile sol-gel method and fast spraying process discovered that by optimizing the ratio of three transition metal nitrates, annealing temperature, and the number of spraying layers, the Solar Selective Absorber Coatings (SSACs) with solar absorptance of 0.93 and vertical emittance of 0.11 were obtained which is good for solar absorber indication. Barati Darband et al. [11] in their study highlighted the usage of CoNiFe alloy as effective and stable electrocatalyst. In this research, self-made Ni-Fe-Co electrode was developed using electrodeposition method. The fabricated electrocatalyst exhibited excellent properties for the evolution of hydrogen and oxygen.

Chaudhary et al. [12] developed ternary Fe-Co-Ni alloy system in search of the next generation rotating electrical machine which possesses high curie temperature, mechanically strong and magnetic soft magnet. The authors discovered that Co-lean alloys exhibited good combination of magnetic, mechanical, and electrical properties but low Curie temperatures. Ledwig et al. [13] in their research of electrodeposited Ni-Co-Fe nanocrystalline coating on copper plate determined the properties required for good-quality MEMS components. Ledwig et al. [13] concluded that Ni-Fe-Co coatings exhibit soft magnetic properties with coercivity below 23 Oe, besides discovering that the corrosion resistance of Ni-Co-Fe coatings is satisfactory with notation that the higher Fe content leads to the deterioration of the corrosion resistance. Ledwig et al. [13] suggested that Ni-Fe-Co coating could be a promising material for MEMS application.

Heat treatment technique is an additional approach for enhancing the performance of the coating. The heating and cooling processes change the composition and structure of metals and alloys [14]. Depending on the materials used and the desired outcomes, various heat treatment processes, such as annealing, tempering, normalizing, and hardening, can increase a material's strength. Heat treatment enhances the strength, flexibility, and toughness of metals and alloys. Arias et al. [15] evaluated the effect of heat treatment on tribological properties of Ni-B coatings on low carbon steel. According to the finding, the hardness and Young Modulus of Ni-B coatings were improved, and the value recorded was 1.6 times higher compared to untreated coatings after implementing heat treatment process, due to grain refinement process during heating.

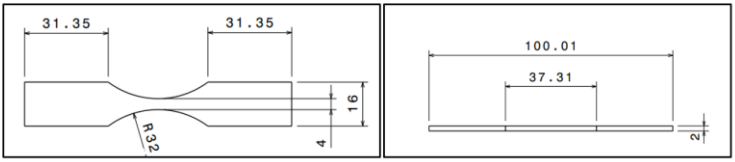
The tensile test, which involves applying a controlled tension to a specimen until failure, is a crucial and common engineering test used for all metallic materials. It provides information about the material's yield strength, elongation at break, ultimate tensile strength, Young's modulus, and other properties [16]. Strain rate, or the rate at which the specimen under test deforms, is a critical tensile test variable that is controlled within specified limits based on the type of test being done.

This paper will assess the surface roughness and hardness, besides conducting tensile test to determine the impact of the Cobalt-Nickel-Iron (CoNiFe) nanocrystalline coating on the tensile performance of coated mild steel, and evaluate the impact of deposition time, varying current and additional heat treatment process on the strength and hardness of coating. It is expected that both yield and ultimate tensile strength of the mild steel substrate will improve with the application of CoNiFe nanocrystalline coating. This may be due to the fact that hard coating could suppress the initiation of cracks; as such, higher stress is needed for crack initiating. During the crack propagation period, the hard coating cracked at a relative higher velocity, which led to the cracking of the ductile substrate and elongation reduction [17]. It is also expected that both surface roughness and microhardness of the mild steel substrate will improve with the application of CoNiFe nanocrystalline coating.

## Methodology

### Sample preparation

Material used for this study was uncoated mild steel substrate. Mild steel plates with 2 mm thickness were subsequently cut into the dimensions of 100 mm x 16 mm using a water jet machine, before further cutting them into dog bone forms with size that adheres to ASTM E466 standard as illustrated in Figures 1a and b. In order to promote uniformity and comparability throughout the studies, the sizes of the samples are kept fixed using water jet cutting machine with the data obtained from CATIA format drawing file (.dxf) for precision cutting process. The usage of water jet cutter also ensures that the cutting process leaves no noticeable flaws in the finished products, which could hinder testing results.




(a) (b)

Figure 1: Dimension of dog bone; (a) front view; and (b) top view

### Bath preparation and electroplating process

In this study, mild steel samples were coated with CoNiFe nanocrystalline coating. Current, electrolyte pH and deposition times were among the manipulated variables tested throughout electrodeposition process.

The solution was prepared through the mixture of several sulphate-based powders such as Cobalt sulphate ($CoSO_4$), Nickel sulphate ($NiSO_4$), Iron sulphate ($FeSO_4$) and ascorbic acid ($C_6 H_8 O_6$). The remaining two powders coming from Boric acid ($H_3 BO_3$) and saccharin ($C_7 H_4 NO_3 S$) were added as pH buffer and grain refinement agent respectively. Table 1 shows the chemical composition used to produce an electrolyte for the coating process. The electrolyte solution was created by mixing the ingredients and heating them to 50±3°C, which was maintained throughout the electrodeposition process. A magnetic stirrer was used to swirl the electrolyte mixture to ensure that a homogenous mixture was obtained. In addition, the solution pH was carefully observed, where potassium hydroxide (KOH) solution was added until pH 3 was reached before starting the electrodeposition process.

Table 1: Chemical composition of electrolyte solution

| 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 ($H_3 BO_3$) | 0.267 |
| Ascorbic Acid ($C_6 H_8 O_6$) | 0.067 |
| Saccharine($C_7 H_5 NO_3 S$) | 0.007 |

Electroplating is a well-known method of depositing metal coatings onto a substrate. The principle behind the electroplating process works by the reaction of metal ions in an acidic electrolyte which comprises a mixture of soluble Co, Ni, and Fe ions inside the similar bath container. For a normal soluble anode, the reaction of metal ions formed is as follows:

$$Co + 2e^- = Co^{2+} \tag{1}$$
$$Ni + 2e^- = Ni^{2+} \tag{2}$$
$$Fe + 2e^- = Fe^{2+} \tag{3}$$

When an insoluble anode (platinum mesh plate) is introduced, oxygen evolves:

$$2H_2O + 4e^- = O_2 + 4H^+ \tag{4}$$

At the cathode, the main reaction is Co, Ni, and Fe deposition, as described by Equations (1), (2), and (3). Hydrogen evolution takes place as a secondary reaction at the cathode surface:

$$2H^+ + 2e^- = H_2 \tag{5}$$

If the electrolyte/electrode movement is limited due to the reduction of metal Ion quantity in the bath, the secondary reaction in Equation (5) reduces current efficiency and local pH, which can result in deposit porosity due to hydrogen gas bubbles sticking to the coating surface [18]. Figure 2 depicts a schematic diagram of the electrodeposition process setup.

Throughout electrodeposition process, mild steel samples were linked to the cathode, while the platinum plate was attached to anode. The current used during electroplating was set to 1.5A and 3.0 A, while the pH used was 3 and 5, respectively. Deposition time of 15, 30, and 45 minutes was used throughout the electrodeposition process. It is critical to monitor the temperature of the electrolyte solution during the electroplating process. Overheating the solution may result in changes of its composition and a decrease in the quality of the coating material deposited on the substrate. Table 2 summarizes the parameters involved in the electrodeposition process.

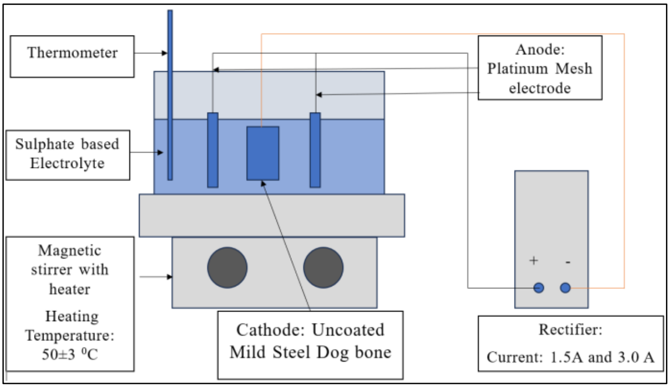



Figure 2: Schematic diagram of the setup for the electrodeposition process

Table 2: Summary of parameters involved in the electrodeposition process

| Parameters | Control setting |
|---|---|
| Arrangement of electrode | Anode: Platinum wire mesh<br>Cathode: Mild steel substate (Dog bone shape) |
| Temperature | 50±3 °C |
| Electrolyte pH | 3 and 5 |
| Current used | 1.5 and 3.0 A |
| Deposition time | 15, 30, and 45 minutes |

### Heat treatment process

The CoNiFe nanocrystalline coated mild steel samples received heat treatment after electrodeposition process. The CoNiFe nanocrystalline coated samples were subjected to a heat treatment procedure that entails immersing them for two hours soaking time at a continuous temperature of 300 °C in a furnace. Following heat treatment, the samples were slowly cooled using a normalizing technique. This approach was chosen because it offers a moderate and steady cooling rate, which reduces the possibility of residual stresses and crack formation. The coating's mechanical and structural qualities were determined after the heat treatment procedure.

## Characterization and testing

All samples were carried out for surface roughness, Vickers microhardness and tensile test. The samples were categorized as presented in Table 3. The surface roughness of the CoNiFe nanocrystalline coating on the mild steel sample was investigated using a 3D surface metrology machine. The coating's surface roughness was influenced by the pH, current, deposition time and heat treatment process. Selecting a number of locations on the coated mild steel surfaces allows the average value of the surface roughness profile, *R* to be calculated. The outcome from surface roughness measurement is to find the lowest possible surface roughness value for the determination of the best coating samples.

Table 3: Summary of samples involved for this study

| Type of samples | Parameters |
|---|---|
| CoNiFe nanocrystalline coating - no heat treatment process | 15-, 30- and 45-minutes deposition time at fixed pH 3; I:1.5 A |
| CoNiFe nanocrystalline coating -with heat treatment process | 15-, 30- and 45-minutes deposition time at fixed pH 3; I:1.5 A |
| CoNiFe nanocrystalline coating -with different pH solution | 30-minutes deposition time, at pH 5; I:1.5 A |
| CoNiFe nanocrystalline coating - with different current setting | 30-minutes deposition time, at pH 3; I:3.0 A |
| uncoated mild steel | Uncoated mild steel-reference sample for comparison |

The Vickers microhardness tester machine, MITUTOYO MVK-H1, was used to conduct microhardness testing as to evaluate the hardness of the CoNiFe nanocrystalline coating of the uncoated mild steel. With a 10 N load, the specimen's surface was indented. An average of five measurements was taken at various locations of the central portion of each specimen's surface to obtain the final microhardness values. The target of Vickers microhardness test is to determine the best hardness coating through the highest microhardness values obtained at the end of measurement.

Tensile tests were carried out to ascertain the mechanical characteristics of the base metal and CoNiFe nanocrystalline coating. The servohydraulic Instron 8801 test apparatus, which is capable of applying axial forces of up to 100 kN, was used for the testing. Specimens were 2 mm thick and had a gauge length of 37.31 mm. Tensile tests were conducted at constant loading rate of 0.5 mm/min until failure. The highest yield strength and ultimate tensile test obtained from tensile test will be the utmost priority for the determination of best coating condition.

## Results and Discussion

### Surface roughness

The outcomes of the surface roughness measurement are covered in this section. In the following sub-section, the impact of surface roughness at various deposition time, heat treatment and electrodeposition parameters such as current and pH are being explored.

The effect of surface roughness of CoNiFe nanocrystalline coating at different deposition time

Figure 3 displays the surface roughness data obtained for mild steel coated with different CoNiFe nanocrystalline deposition time. The surface roughness value decreases as the deposition time increases from 15 minutes to 45 minutes. Reduction of surface roughness value indicates a sign of improvement through the introduction of CoNiFe nanocrystalline coating onto the surface of uncoated mild steel. The highest surface roughness result obtained for uncoated mild steel was 4.233 µm, while mild steel coated for 45 minutes CoNiFe nanocrystalline coating recorded the lowest surface roughness of 2.354 µm. Mild steel samples coated for 15 and 30 minutes recorded the surface roughness of 3.737 µm and 2.667 µm, respectively. From the results, it can be deduced that the surface roughness is affected by the duration of deposition time, due to void formation and oxidation process that might take place on the surface material [19].

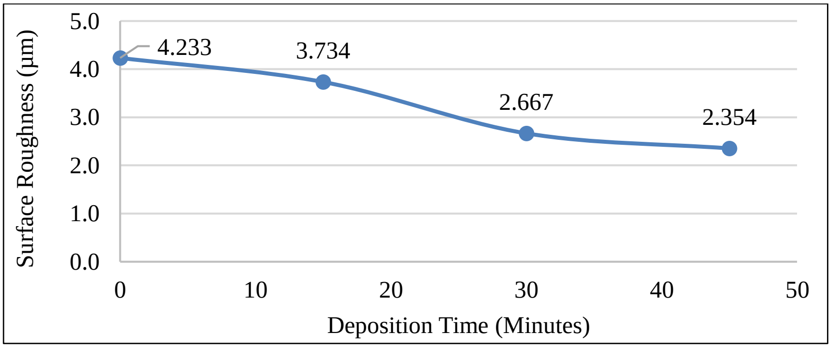



Figure 3: Comparison of CoNiFe nanocrystalline surface roughness electrodeposited at different deposition time

The presence of oxidation and corrosion initially produces an increase in the surface roughness of uncoated mild steel. Exposure to moisture, air, or severe circumstances without a protective coating lead to higher susceptibility of oxidation and corrosion on the uncoated mild steel. Iron oxide (rust) can form on the surface as a result of oxidation, increasing the surface roughness value. However, the smoothing effect was established as the surface roughness of CoNiFe nanocrystalline coating reached the lowest after 45 minutes deposition time. Coating is a common method of giving surfaces a smoother, more uniform appearance [20]. Surface roughness can be reduced by filling up or bridging microscopic surface flaws using these coatings. Furthermore, coatings can improve the overall surface smoothness by concealing flaws and voids, besides providing a more polished surface.

## The effect of surface roughness of CoNiFe nanocrystalline coating with or without heat treatment process

Figure 4 shows the comparison of CoNiFe nanocrystalline coating surface roughness for both CoNiFe electrodeposited coating and CoNiFe electrodeposited coating with heat treatment. CoNiFe nanocrystalline coatings for both electrodeposited samples and heat treatment samples were compared for 15, 30 and 45 minutes. The surface roughness of CoNiFe nanocrystalline coating for both electrodeposited samples and heat treatment samples showed similar decreasing pattern. The highest surface roughness recorded for both CoNiFe nanocrystalline coating process at 15 minutes deposition time was 3.740 μm and 3.590 μm, respectively. Meanwhile, the lowest surface roughness obtained from both samples with and without heat treatment process at 45 minutes deposition time was 2.350 μm and 1.820 μm respectively.

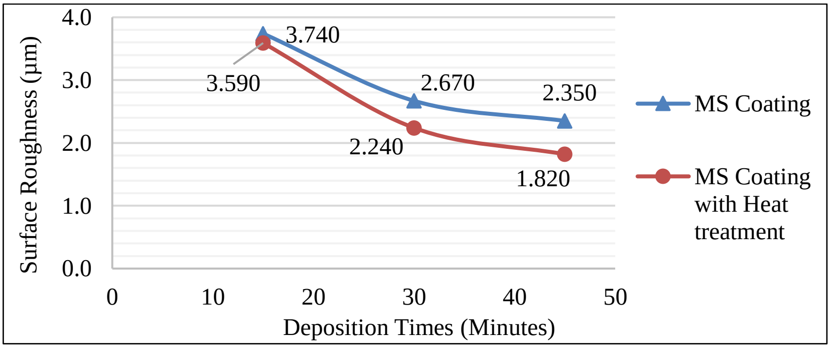



Figure 4: Surface roughness comparison for CoNiFe nanocrystalline coating with and without heat treatment process

Heat treatment was assumed to have increased the particle compactness and density of CoNiFe nanocrystalline coating. The levelling and smoothing impact of heat treatment on the coating confirms this trend. Throughout the heat treatment, the CoNiFe nanocrystalline coating is subjected to controlled heating and cooling cycles. Heat treatment process promotes the redistribution and relaxation of tensions within the coating, improving adhesion and reducing surface defects. The heat treatment process also improves the flow and levelling of the coating material, resulting in a smoother surface with decreased surface roughness value [21]. The linear decrease in surface roughness is observed as heat treatment process allows the coating material to reflow and rearrange, minimizing surface voids and inconsistencies. The regulated heat application causes structural changes in the coating, resulting in a more uniform surface texture. Furthermore, the decrease in surface roughness is also attributed to the creation of early clusters with smaller particle sizes and dense populations as nucleation density increases [22].

### The effect of surface roughness of CoNiFe nanocrystalline coating with varying coating parameters at constant deposition time (current, pH, and heat treatment process)

The surface roughness obtained for mild steel coated with 30 minutes deposition times at various pH and current is shown in Figure 5. Based on the findings, it is worth noting that the surface roughness value increased as the pH value increased. The surface roughness of mild steel CoNiFe nanocrystalline coating with 30 minutes deposition time (pH: 5 and current of 1.5 A) was higher than that of coated mild steel with 30 minutes deposition time (pH: 3 and current of 1.5 A).

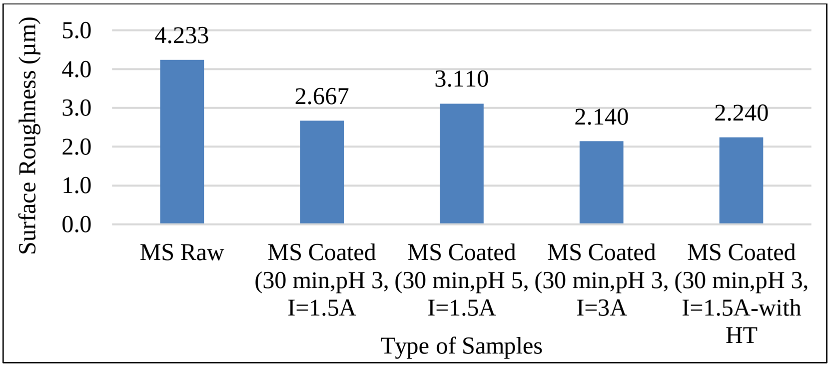



Figure 5: Surface roughness comparison of CoNiFe nanocrystalline coated for 30 minutes with different parameters

At the same current setting, less coating material was deposited onto the surface since higher electrolyte pH value (pH=5) contributes to a lower deposition rate. As a result, the coating thickness might be reduced, and surface roughness may not be filled or levelled out effectively which leads to higher surface roughness. Resali et al. [23] discovered that the effect of phase formation on particle size was insignificant. However, as the pH increased, the nucleation of crystallites resulted in the formation of coalesced particles.

Hence, the particle tended to aggregate and produced larger particles, while void formation was discovered in samples prepared at higher bath pH.

It is also worth noting that as the applied current increased from 1.5 A to 3.0 A, the surface roughness decreased. The decrease in surface roughness might be due to a higher current applied during electrodeposition process, which results in a faster deposition rate. A thicker and more uniform coating occurred on the mild steel surface due to the faster deposition rate [24]. The faster deposition process was able to aid in the filling of surface imperfections and the reduction of surface roughness. The addition of heat treatment process on the CoNiFe nanocrystalline coating also effectively reduced the surface roughness compared to the other 30 minutes CoNiFe nanocrystalline coating. Similar improvement of surface roughness was discovered by Bejaxhin et al. [25]. They observed an increase in hardness value due to the heat treatment effect. They also claimed that 41% improvement in surface roughness was obtained by the effect of heat treatment as well as the specific machining conditions.

## Microhardness test

In the following subsection, the microhardness impacts of CoNiFe nanocrystalline coating at various deposition durations, heat treatment processes, and parameters such as current and pH are discussed.

### The effect of CoNiFe nanocrystalline microhardness at different deposition times

Figure 6 depicts the change in hardness qualities as a function of deposition time at 15 minutes, 30 minutes, and 45 minutes for each sample. Mild steel has a rapid increase in hardness from 171.44 HV (uncoated mild steel sample) to 245.36 HV (15 minutes deposition times), and a sustained increasing pattern of 252.56 HV and 267.46 HV for coating times of 30 minutes and 45 minutes, respectively.

The steady increase in hardness from uncoated mild steel to mild steel with 45 minutes deposition time may be attributed to the changes in the phase structure, effect of porosity, solid hardening mechanism and particle size of the nanocrystalline deposit [26]. Longer deposition time also enables more CoNiFe nanocrystalline coating to be deposited on the mild steel substrate, resulting in a thicker covering which makes the surface of the coating harder [23]. As particle size decreases, the microstructure becomes denser with particle barriers and grain boundaries [24]. During electrodeposition process, metal ions from the electrolyte may reduce and deposit onto the substrate, resulting in metal grains. Longer deposition time give grains more time to develop and mature.  Smaller grains have a higher dislocation density, which contributes to the high microhardness value. Dislocations are crystal structural imperfections that limit dislocation mobility while also strengthening the material.

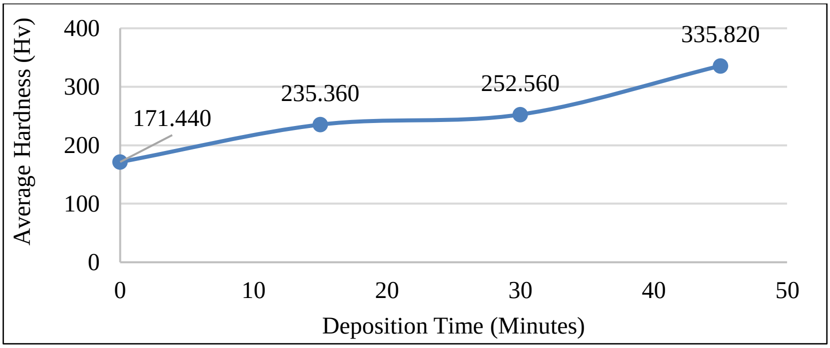



Figure 6: Vickers microhardness test for CoNiFe nanocrystalline coating at different deposition time

## The effect of CoNiFe nanocrystalline microhardness with or without heat treatment process

The microhardness of CoNiFe nanocrystalline coated surfaces with and without heat treatment is shown in Figure 7. The results were plotted, and both CoNiFe nanocrystalline coating with and without heat treatment process exhibited an increase in microhardness. It can be seen that CoNiFe nanocrystalline coating with additional heat treatment process had a higher hardness as compared to CoNiFe nanocrystalline coating without heat treatment process.

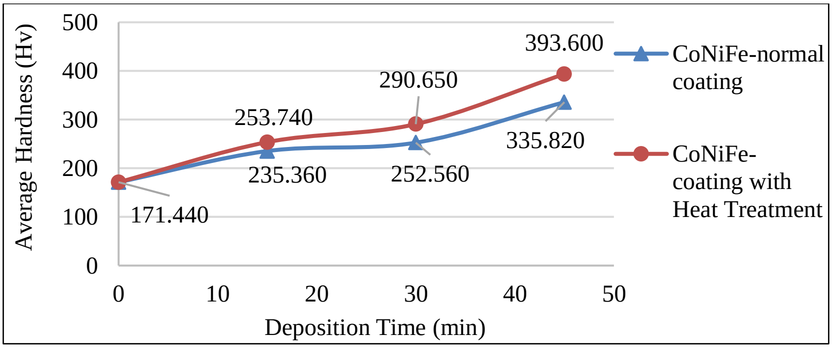



Figure 7: Vickers hardness test comparison for CoNiFe nanocrystalline coating at different deposition time (with or without heat treatment process)

The microhardness value for CoNiFe nanocrystalline coating with heat treatment process was 393.6 HV (45 minutes deposition time) and the lowest average microhardness CoNiFe nanocrystalline coating with heat treatment process was 253.74 HV (15 minutes deposition time). In comparison, the highest microhardness value for CoNiFe nanocrystalline coating without heat treatment was 335.82 HV for the same deposition duration (45 minutes deposition time) and the lowest microhardness CoNiFe nanocrystalline coating without heat treatment process was 235.36 HV (15 minutes deposition time).

The data clearly showed that CoNiFe nanocrystalline coating with heat treatment process surpassed the hardness performance of CoNiFe nanocrystalline coating without heat treatment process. Heat treatment produces grain development and coarsening in the coating and substrate, enhancing their mechanical properties. As grain size grows, the number of grain boundaries per unit volume reduces, and the remaining grain boundaries become more resistant to dislocation movement. Particle barriers also restrict dislocation motion and contribute to the formation of a more complex layer in the microstructure [27]. Furthermore, the homogeneous particle distribution, compact and dense microstructure, as well as higher temperature resulted in CoNiFe nanocrystalline coating with a much better microhardness [28]. Overall, the application of CoNiFe nanocrystalline coating with and without heat treatment process improves the hardness performance of uncoated mild steel substrate.

### The effect of CoNiFe nanocrystalline microhardness when varying coating parameters at constant deposition time (current, pH, and heat treatment process)

The average value of microhardness obtained for mild steel using the same deposition time and various pH and current value is shown in Figure 8. It can be seen that the microhardness value decreased as the pH value for the constant 30 minutes deposition time increased from pH 3 to 5. The microhardness of mild steel CoNiFe nanocrystalline coating with 30 minutes deposition time (pH: 5 and current of 1.5 A) was lower than that of coated mild steel with 30 minutes deposition time (pH: 3 and current of 1.5 A). The decreasing pattern of microhardness from 252.56 HV to 212.36 HV may be related to changes in coating structure and properties as the pH shifts from 3 to 5. At pH 3, the electrodeposition reaction kinetics may be more favourable, resulting in a more dense and compact coating.

It is well known that the performance of material hardness is usually in excellent form if its particle size is smaller. However, at pH 5, the electrodeposition process may be less effective and form bigger particle size, resulting in a thinner layer with agglomeration and void formation that may happen in the microstructure [23]. These structural changes may result in a decrease of microhardness at pH 5. It is also worth noticing that the microhardness value improved as the applied current increased from 1.5 A to 3.0 A. The rising trend in average microhardness is caused by a larger current delivered during the electrodeposition process, which results in a faster deposition rate and may promote the nucleation and growth of smaller grains [24].

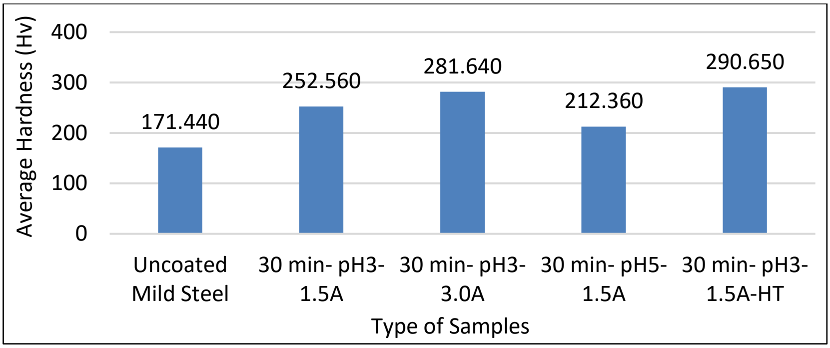



Figure 8: Vickers hardness test comparison for CoNiFe nanocrystalline coating fixed at 30 minutes deposition time (varies pH, current and heat treatment)

Smaller grains have higher dislocation density, which limits dislocation movement and increases microhardness [27]. Improved grain refining at a current intensity of 3 A versus 1.5 A may result in higher microhardness values. The addition of the heat treatment process raises the microhardness compared to sample coated for 30 minutes deposition time. The synergistic effect between nanoparticles introduction and microstructural features on coating microhardness results in higher values of CoNiFe nanocrystalline coating coated for 30 minutes deposition time with additional heat treatment process as compared to their standard counterpart, with a progressive hardness increase of up to 290.65 HV. Addition of heat treatment given to as-deposited CoNiFe nanocrystalline effectively strengthens coatings hardness. The findings were also reported by Pedrizzetti et al. [29] who discovered the synergistic effect between nanoparticles introduction and microstructural features on coating microhardness which resulted in higher hardness values for nanocomposites compared to their standard counterpart, with a progressive hardness increase of up to heat treatment at 400 °C.

## Tensile performance

This section discusses the tensile performance at various deposition time, additional heat treatment process, and various electrodeposition parameters such as current and pH.

## Tensile performance of CoNiFe nanocrystalline at different deposition time

The stress-strain curves of all specimens are shown in Figure 9. The ultimate tensile strength of coated mild steel was seen to be higher than uncoated mild steel, as shown in Table 4.

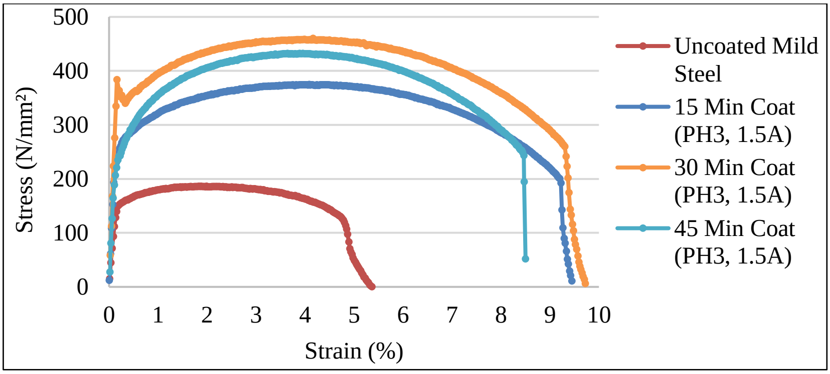



Figure 9: Stress-strain curves of CoNiFe nanocrystalline coating applied at different deposition time

Table 4: The yield stress and ultimate strength of uncoated and CoNiFe nanocrystalline coated specimens at different deposition time

| Specimen | Yield stress $\sigma_y$, MPa | Ultimate Tensile strength $\sigma_u$, MPa |
|---|---|---|
| Uncoated | 149.400 | 186.780 |
| 15 min | 267.310 | 375.920 |
| 30 min | 384.060 | 460.380 |
| 45 min | 243.830 | 432.670 |

Uncoated mild steel, mild steel coated for 15 minutes, 30 minutes, and 45 minutes have ultimate tensile strengths of 186.780 MPa, 375.920 MPa, 460.380 MPa, and 432.670 MPa, respectively. The fact that CoNiFe nanocrystalline coated mild steel had a greater ultimate tensile strength than uncoated mild steel suggests that the coating implementation improved the mechanical properties of mild steel substrate [30]. The uncoated mild steel appeared to have gained strength as a result of the coating, allowing them to withstand greater quantities of stress before failure [31]. However, the ultimate tensile strength of 30 minutes was greater than that of 45 minutes. This scenario may be explained by factors such as coating quality and possibly due to the material experiencing transformations or alterations that modify its mechanical properties.

The coating's quality may also have an effect on its final tensile strength. Uniformity, adherence, presents of voids, agglomeration and abnormalities on the coating layer can all have a significant impact on the mechanical properties of CoNiFe nanocrystalline coated surfaces [32]. Furthermore, the yield stress for uncoated, 15 minutes, 30 minutes, and 45 minutes CoNiFe nanocrystalline coating was 149.400 MPa, 267.310 MPa, 384.060 MPa, and 243.830 MPa, respectively which showed similar increasing pattern as the ultimate tensile strength measurement.

## Tensile performance of CoNiFe nanocrystalline with and without heat treatment process

Figure 10 shows the ultimate tensile strength values of CoNiFe nanocrystalline coating samples subjected to different heat treatment durations compared to CoNiFe nanocrystalline samples. The ultimate tensile strength values for CoNiFe nanocrystalline coating with heat treatment varied according to the deposition time, as shown in Table 5 where the values were 407.330 MPa at 15 minutes, 467.670 MPa at 30 minutes, and 559.110 MPa at 45 minutes. In contrast, recorded ultimate tensile stress for as-coated CoNiFe nanocrystalline samples was 375.920 at 15 minutes, 460.380 MPa at 30 minutes, and 432.67 0 MPa at 45 minutes which were significantly lower as compared to the heat-treated coatings.

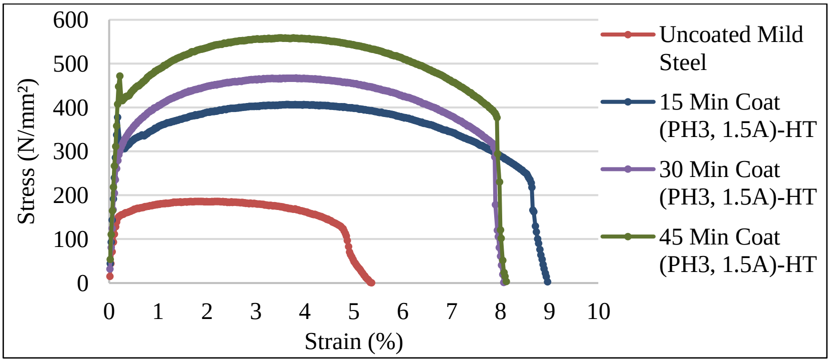



Figure 10: Stress-strain curves of CoNiFe nanocrystalline coating applied at different deposition time with heat treatment process

According to the results, heat treatment has a significant impact on the ultimate tensile strength of CoNiFe nanocrystalline coated materials and affects the material's microstructure, resulting in variations in strength. These observations could be linked to the formation of soft ferrite during the cooling process [33]. The ultimate tensile strength of CoNiFe nanocrystalline coated was much higher after the coating underwent heat treatment of minimal deposition time and additional heat treatment process enhanced with mechanical strength which were represented by the higher ultimate tensile strength and yield stress compared to uncoated sample. The continuous trend of ultimate tensile strength after 30 and 45 minutes CoNiFe nanocrystalline coating with heat treatment could be attributed to other microstructural changes, such as precipitation hardening or grain refinement, which contributed to higher mechanical strength [34].

Table 5: The yield stress and ultimate strength of CoNiFe coated specimens with heat treatment process

| Specimen | Yield stress $\sigma_y$, MPa | Ultimate strength $\sigma_u$, MPa |
|---|---|---|
| 15 min | 378.350 | 407.330 |
| 30 min | 301.200 | 467.670 |
| 45 min | 472.350 | 559.110 |

Despite the fact that additional heat treatment process increased the ultimate tensile strength of CoNiFe nanocrystalline coating, the resulting yield strength did not follow the same increment pattern. The yield stress of CoNiFe nanocrystalline coating coated for 45 minutes deposition time was found to be the highest (472.350 MPa) whereas 30 minutes coating sample showed the lowest yield stress (301.200 MPa). During heat treatment procedure, changes in the microstructure of the CoNiFe nanocrystalline coating may occur, possibly contributing to a slight decline in yield stress for the 30 minutes coating sample. Surprisingly, the yield stress increased dramatically after 45 minutes of heat treatment (472.350 MPa).

## Tensile performance of CoNiFe nanocrystalline when varying coating parameters at constant deposition time (current, pH, heat treatment process)

Figure 11 depicts the tensile test data which demonstrate the ultimate tensile strength of mild steel when coated under various conditions. According to Table 6, the ultimate tensile strength of CoNiFe nanocrystalline coating greatly increased when the current intensity was increased at a constant pH (520.590 MPa). The result demonstrated that the tensile properties of the CoNiFe nanocrystalline coating were determined by the current adjustment factor in the electrodeposition process. The rise in the ultimate tensile strength was attributed to different strengthening mechanisms, such as solid solution and precipitation strengthening [23]. The strongly adhesive CoNiFe nanocrystalline covering might effectively suppress the failure of the mild steel substrate at early stages. PJ Teng et al. also discovered that the coating layer significantly improved the tensile resistance of the substrate and concluded that the tensile properties of their coated specimens increased due to the increase in current densities throughout electrodeposition process [35].

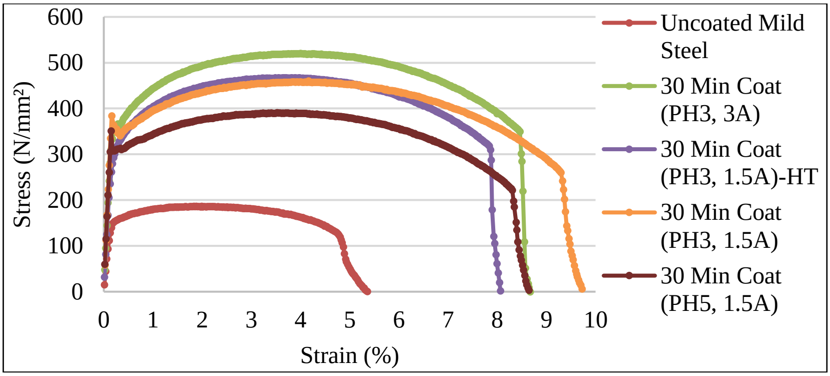



Figure 11: Stress-strain curves of CoNiFe nanocrystalline coating applied at 30 minutes deposition time with various parameter setup (different pH, current and heat treatment)

Table 6: The yield stress and ultimate strength of heat treated CoNiFe coated specimens

| Specimen | Yield stress σy, MPa | Ultimate strength σu, MPa |
|---|---|---|
| Uncoated | 149.40 | 186.78 |
| 30 min (pH=3, I=1.5 A)-HT | 301.21 | 467.71 |
| 30 min (pH=3, I=1.5 A) | 384.06 | 460.38 |
| 30 min (pH=3, I=3 A) | 348.19 | 520.59 |
| 30 min (pH=5, I=1.5 A) | 351.23 | 390.99 |

However, when the pH of the electrolyte was increased to 5 with constant current intensity of 1.5 A, the ultimate tensile strength slightly reduced to 390.99 MPa. This demonstrated that, as compared to the effects of current intensity, a higher pH value has a lesser effect on ultimate tensile stress. These data suggested that the intensity of the current and the pH level during the coating process could have a significant impact on the ultimate tensile stress of CoNiFe nanocrystalline coating. This finding was similar to M. Stamenovic et al. who reported that alkaline solutions at higher pH value caused a decrease on the tensile properties. During the treatment of the samples, they coated the inner surfaces and went deeper into the samples through micro-cracks and other surface damages which existed after fabrication and shrinkage of the material. On the other hand, the composite material was strengthened during treatment of samples with acids (lower pH) and increment in tensile properties was obtained [36].

## Conclusions

It is concluded that surface roughness, Vickers hardness and tensile enhancement are experienced throughout the introduction of CoNiFe nanocrystalline coating. The highest improvement for surface roughness, microhardness and tensile performance was recorded by CoNiFe nanocrystalline coating at 45 minutes deposition time with pH 3, I:1.5 A and heat treated (surface roughness: 1.82 µm; microhardness: 393.6 Hv; YS: 472.35 MPa; UTS: 186.78 MPa) as compared to reference uncoated mild steel (surface roughness:4.233 µm; microhardness: 171.44 Hv; YS: 149.40 MPa; UTS: 186.78 MPa). It is shown that both surface roughness and hardness obtained are 2.3 times better than the reference uncoated mild steel. Both yield strength and ultimate tensile strength values show a direct improvement of 3.16 and 3 times compared to uncoated mild steel samples. The introduction of higher current setting boosts up both yield strength (348.19 MPa) and ultimate tensile strength (520.59 MPa). CoNiFe nanocrystalline coating successfully serves as a protective layer and performance booster in which it protects the underlying mild steel from external forces that can cause failure of low strength purpose.

## 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.

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## Conflict of Interests

All authors declare that they have no conflicts of interest.

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