

Understanding of Heat Treatment on the Durability of Cobalt Alloy Thin Film Coated on Mild Steel

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ABSTRACT

Mild steel is used extensively in infrastructure and industry due to its excellent mechanical quality. However, this type of steel is susceptible to fatigue failure, such as low-high cycle fatigue, thermal fatigue, and contact fatigue, which reduces the life span of components. The objective of the study is to investigate the effect of heat treatments on the CoNiFe coated mild steel on hardness, surface roughness, and fatigue life. The mild steel substrate was electrodeposited using CoNiFe nanoparticles, followed by heat treatment processes such as annealing and quenching. The highest coating mass recorded by 45 min coating without Heat Treatment (HT) was 1.02 g, while the lowest coating mass recorded by 15 min coating without HT was 0.35 g. The highest improvement of microhardness was observed on a 45 min coating without HT, which was 315.82 HV as compared to the uncoated substrate, which was 132.40 HV. The lowest surface roughness was obtained by the 15 min samples without HT (0.20 μm), while the highest recorded surface roughness was obtained on the uncoated substrate (0.42 μm). The fatigue performance of 30 min coating with HT (quenching) was the highest, with a cycle life of 1,000,000 cycles and 80% Ultimate Tensile Strength (UTS) loading, respectively. The lowest fatigue performance was recorded from the uncoated substrate with cycle life and yield stress percentages of 1,450 cycles and 80% of UTS loading, respectively. Coated substrates show significant improvement on fatigue life, indicating the synergy of coating with post heat treatments to enhance the materials durability under cyclic hence showing high potential of improving mild steel's performance for a wide range of applications.

INTRODUCTION

Mild steel has become one of the most widely utilized materials in engineering industries. It is globally used in the development of constructions, roadways and appliances. Mild steel highlights its versatility and

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indicating numerous additional uses (Kareem, 2013). Mild steel has good mechanical qualities, such as durability and toughness against impact and shock loading, but repeated loading can cause wear and fatigue. Fatigue is a failure phenomenon in which material is failing due to repeated cyclic loading before it reaches its ultimate stress, which does not easily occur in a single occurrence (Kaimkuriya et al., 2024). It also refers to alterations of metallic material properties caused by repeated applied cyclic stress and strain, which will lead to cracking and failure (Padzi et al., 2014). Components made from mild steel material are often subjected to cyclic loading, such as fatigue, or environmental effects such as corrosion. Therefore, it is important to examine the influence of cyclic loading and the associated effects on the mechanical properties of mild steel to ensure safety during its desired application. The component will fail if the external stress is greater than the maximum compressive and shear stress. Microcracks are mostly caused and spread by cyclical loading, which leads to failure. Material imperfections like inclusions, voids, and porosities serve as catalysts for fatigue cracks to form (Kaimkuriya et al., 2024). The effect of coatings on fatigue strength of metallic materials has been the subject of research in recent years (Chandra et al., 2020; Ham et al., 2021). Most of these works concentrate on the effect of residual stress in the coating on the fatigue behaviour of materials. In order to improve the fatigue properties, mild steel material needs to undergo a process of improving mechanical properties in order to extend its fatigue life cycle, such as surface coating, thermal treatments, and mechanical treatments (Mirghaderi et al., 2021).

Surface protective coatings have been used in several engineering applications to protect and prolong the lifespan of parts or surfaces exposed to a corrosive environment (Zabri et al., 2022). In order to build, operate, and protect structural systems, construction materials, and machinery in harsh environments, nanoparticle alloy coatings with excellent mechanical and corrosion resistance are needed (Md. Nor et al., 2019). Electrodeposition is one of the methods used to create metallic coatings on a substrate through the adjustment of potential and current. The electroplated layers are anticipated to have a strong adhesion, glassy appearance, and fine-grained structure (Cattaneo & Riegel, 2025). Several researchers discovered that depositing alloy coating using the electrodeposition method on the steel surface provides superior coatings with a smooth surface finish, increases resistance against corrosion, and enhances mechanical qualities, including ductility, surface hardness, strength, and wear resistance, as well as aesthetic appearance and qualities (Fayomi & Popoola, 2012; Tsyntaru, 2016; Karakurkchi et al., 2016). Kumar et al. (2004) found that mild steel electrodeposited with Co-Cr₂O₃ electrolytes increases its surface properties, and cobalt mixture strives better at high temperature.

Co, Ni, and Fe nanoparticles in combination is utilized for a variety of applications. Yang et al. (2021) discovered that by optimizing the ratio of three transition metal nitrates, annealing temperature, and the number of spraying layers on Co-Ni and Fe based metal, Solar Selective Absorber Coatings (SSACs) with vertical emittance of 0.11 and solar absorptance of 0.93 were achieved, which is good for solar absorber indication. Chaudhary et al. (2021) created a ternary FeCoNi alloy system in search of the next generation rotating electrical machine with a high Curie temperature, magnetic soft magnet, and mechanical strength. The scientists discovered that Co-lean alloys had a favorable combination of magnetic, electrical, and mechanical capability, aside from low Curie temperatures. Ledwig et al. (2021) identified the qualities required for high-quality Micro-Electromechanical System (MEMS) components throughout the study of electrodeposited Ni-Co-Fe nanocrystalline coating on copper plate. Ledwig et al. (2021) concluded that Ni-Fe-Co coatings display soft magnetic characteristics with coercivity less than 23 Oe, as well as discovering that the corrosion resistance of Ni-Co-Fe coatings is good, with the exception that increasing Fe content leads to a decrease in corrosion resistance. Thus, the authors reported that Ni-Fe-Co coating would be a promising material for MEMS applications. Zabri et al. (2023) found that 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:1.5A and heat treated (surface roughness: 1.82 μm ; microhardness: 393.6 HV; YS: 472.35 MPa; UTS: 559.11 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). Both surface roughness and hardness obtained from cobalt alloy nanoparticle coatings are 2.3 times better than

the uncoated mild steel. Zabri et al. (2023) also concluded that the yield strength and ultimate tensile strength of cobalt alloy nanoparticle coatings show improvement of 3.16 and 3 times compared to uncoated mild steel samples.

Heat treatment is the process of heating and cooling an alloy or metal in order to achieve the required mechanical qualities such as hardness, toughness, Young's modulus, yield strength, ultimate tensile strength, elongation, and percentage decrease (Kandpal et al., 2021). Younan et al. (2002) also mentioned that the process of annealing shows a relationship between microhardness and structural changes in Ni-Co-P deposits during annealing. Three factors that impact the microhardness which is grain size, density, and impurity-induced dislocations. Deposition with smaller particles and greater stresses has higher microhardness. The observed temperature spike at 400 °C is caused by the synthesis of small particles, the inclusion of cobalt atoms in the nickel lattice, and the generation of a harder Ni_3P phase. In general, structural alterations brought about by annealing influence the microhardness of Ni-Co-P deposits. Induction heating and quenching process hardens the surface layer of structural steel materials, besides giving the coated surface a higher compressive residual stress, which improves fatigue characteristics of materials (Komotori et al., 2001). Introduction of annealing can prolong the crack incubation time, besides helping to extend the fatigue life of the mild steel (Du et al., 2004).

This paper will assess the coating mass trend, coating hardness, surface roughness as well as conducting fatigue test to determine the impact of the Cobalt-Nickel-Iron (CoNiFe) nanoparticle coating on the mechanical performance of coated mild steel substrates, and evaluate the impact of deposition time, and additional heat treatment process on the mass, roughness, hardness and maximum cycle of the applied coating. It is expected that both cycle life and maximum strength of the mild steel substrates will improve with the application of CoNiFe nanoparticles coating. The hard coating could suppress the initiation of cracks to enable a higher stress tolerance before the initial crack and crack propagation. It is also expected that both surface roughness and microhardness of the mild steel substrates will improve with the application of CoNiFe nanoparticle coating.

METHODOLOGY

Substrate Preparation

Materials used for this work were an uncoated mild steel plate with specification and grade of MS EN 10025-2:2011 S275JR+AR. The 3 mm thick mild steel plate was subsequently cut into a dog bone shape that adheres to the ASTM E466 standard as shown in Fig 1. In order to practice accuracy and precision throughout the work, the sizes of the substrate were kept constant using a water jet cutting machine.

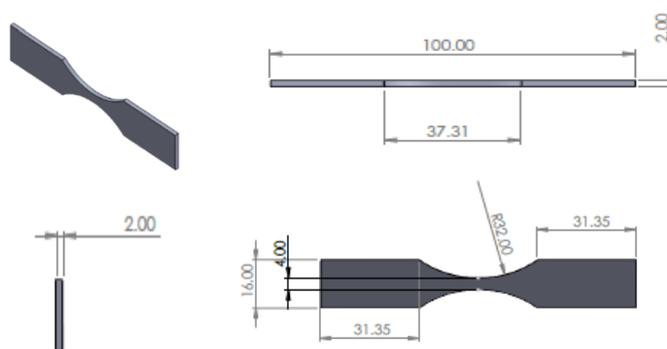


Fig. 1. Dog bone samples prepared using water jet cutter, dimension in unit mm.

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Prior to the cutting process, all uncoated mild steel substrates were subjected to an extensive cleaning process until the substrate surfaces were clean of the oxide layer. This cleaning process required the usage of a steel brush followed by 120, 240, and 320 grit flap disks. The samples were then cleaned using distilled water and rinsed with ethanol. This step-by-step approach ensured that the surfaces of the specimens were uniformly clean and smooth before proceeding to the coating process.

Electrolyte Preparation and Electrodeposition Process

Mild steel substrates were coated using a sulphate-based solution. Table 1 shows the chemical powders used for preparing the Cobalt alloy coating electrolytes. The electrolyte was prepared from Cobalt sulphate (CoSO_4), Nickel sulphate (NiSO_4), Iron sulphate (FeSO_4), and ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) using the amounts stated in Table 1. Additional powders coming from saccharin ($\text{C}_7\text{H}_4\text{NO}_3\text{S}$) and Boric acid (H_3BO_3) served as grain refinement agents and pH buffers, respectively. pH buffers help to stabilize and retain the solution's pH, which is crucial for consistent coating quality and chemical stability during the process. The usage of Saccharine as surface refinement also contributes in achieving a smoother and more uniform coating on the steel samples. In addition, the solution pH was carefully set, where 25 ml of 1.0695 molar (M) potassium hydroxide (KOH) solution was added until pH 3 was reached before starting the electrodeposition process. The samples for the electroplating process adhere to ASTM B183-18. The coating parameters applied were scaled up accordingly, with 1 litre of electrolyte, based on optimization from previous studies (Hyie et al., 2016).

Table 1. Chemical powders used for preparing coating electrolytes

Compound	No. of moles	Mass (g)
Cobalt Sulphate (CoSO_4)	0.050	14.054
Nickel Sulphate (NiSO_4)	0.133	35.048
Iron (II) Sulphate (FeSO_4)	0.020	5.560
Boric acid (H_3BO_3)	0.267	16.488
Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$)	0.067	11.737
Saccharine ($\text{C}_7\text{H}_5\text{NO}_3\text{S}$)	0.007	1.367

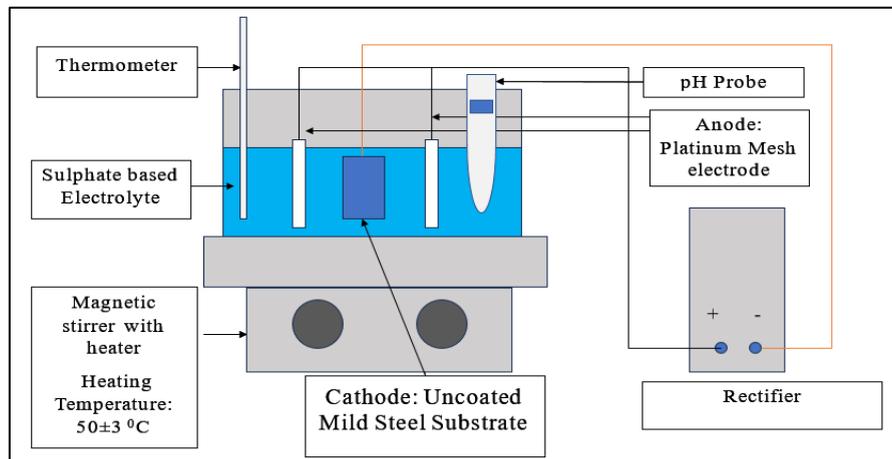


Fig. 2. Schematic setup for coating process using electrodeposition method.

Throughout the electrodeposition process, platinum plates were linked to the anode while a mild steel substrate was attached to the cathode. The current during electroplating was set to 1.5 A, while the pH was set constant at pH 3. Deposition times of 15 min, 30 min, and 45 min were used throughout the

electrodeposition process. The different selection of deposition time was carried out to analyse the effect of different coating deposition times on the surface roughness, hardness, and overall fatigue performance of coated mild steel. Fig 2 shows the schematic setup for the coating process using the electrodeposition method. Table 2 depicts the set of parameters used for the electrodeposition process.

Table 2. A set of parameters used for the electrodeposition process

Parameters	Control setting
Arrangement of electrode	Cathode: Mild steel substrate (Dog bone shape) Anode: Platinum wire mesh
Temperature	50 ± 5 °C
Electrolyte pH	3
Current used	1.5 A
Deposition time	15 min, 30 min, and 45 min

Heat Treatment Process

The coated mild steel substrates were subjected to several heat treatment processes, such as annealing and quenching. Annealing is used to reduce internal tensions caused by previous processing through furnace cooling to soften the substrate and increase its ductility, while the goal of quenching is to quickly cool steel from a high temperature to increase its hardness and strength. Both treatments were selected to compare the difference in fatigue performance with the expectation that annealing the substrate could enhance the microstructure, resulting in better formability and machinability (Hernandez-Duran et al., 2021). As for quenching, it is expected that quenching the substrate could increase mechanical strength and wear resistance by converting the microstructure into the hard, brittle phase of martensite (Hernandez-Duran et al., 2021).

The Nabertherm brand furnace was used to conduct the heat treatment process. Table 3 shows the category of samples that have been prepared. Samples were placed in the furnace and heated up to 400 °C with a step temperature of 5 °C per minute. The temperature was kept constant for two hours with a soaking temperature of 400 °C. Maintaining the soaking temperature at 400 °C keeps it below the recrystallisation threshold of steel, preventing unwanted grain formation. Xue et al. (2021) concluded that the absence of recrystallized refinement grains indicated that recovery, rather than recrystallization, occurred at an annealing temperature equal to or lower than 400 °C. The usage of lower temperature could still cater the reduction of internal stress and the stabilization of the coating's microstructure, without reaching the recrystallisation threshold of steel and resulting in improved mechanical characteristics. Afterwards, some of the samples were left in the furnace to cool slowly, while the remaining samples were taken out of the furnace after 2 hours of soaking time had ended to carry out the quenching process by dipping the coated substrates inside the water to obtain rapidly cooled coated substrates.

Table 3. Summary of samples

Type of samples	Parameters
Uncoated mild steel (3 samples)	Run at 90%, 80% and 50% of UTS loading Uncoated mild steel- reference sample for comparison
CoNiFe nanocrystalline coating -with no heat treatment process (4 samples)	15 minutes deposition time at fixed pH 3; I: 1.5 A 30 minutes deposition time at fixed pH 3; I: 1.5 A 30 minutes deposition time at fixed pH 3; I: 1.5 A 45 minutes deposition time at fixed pH 3; I: 1.5 A run at 80% of UTS loading
CoNiFe nanocrystalline coating -with heat treatment process (annealing only) (2 samples)	30 minutes deposition time @pH 3; I: 1.5 A - run at 80% of UTS loading 30 minutes deposition time @pH 3; I: 1.5 A - run at 90% of UTS loading
CoNiFe nanocrystalline coating -with heat treatment process (quenching only) (1 sample)	30 minutes deposition time @pH 3; I: 1.5 A

Characterization and Testing

Coating mass measurement

The coating mass measurements were conducted using a Shimadzu weighing balance with three (3) decimal digit measurements. The mass of all uncoated substrates was measured before coating, after coating, and after the heat treatment process. All measurement was repeated three times, and the average value was taken to ensure accurate and reliable results.

Vickers hardness

The microhardness test was conducted using a hardness testing machine (Mitutoyo MVK-H1), applying an indenter load of 1000 grams using a diamond indenter. The indentation was made on three (3) different locations on each of the substrates to ensure accurate and reliable results.

Surface roughness

The surface roughness test was conducted using a Surf test (Mitutoyo SV-600) equipped with a diamond indenter. In order to promote accurate and reliable results, the tests were repeated three (3) times at different locations on the substrate surface. The coating surface roughness was influenced by the deposition time and heat treatment process. Selecting three numbers of locations on the coated mild steel surfaces allows the average value of the surface roughness profile, R_a 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 substrates.

Fatigue test

Axial fatigue tests were carried out conforming to ASTM E466 standard using Shimadzu servo hydraulic machine on mild steel coated and uncoated substrates. These tests were done under constant amplitude sinusoidal cyclic loading with a load ratio (LR) ($R = S_{min}/S_{max}$) of 0.05 and loading frequency of 5 Hz under room temperature. These tests were conducted at various maximum stress levels (S_{max}), starting at 90%, 80%, and 50% of the material's ultimate tensile strength (UTS). Three coated mild steel substrates with post coating treatment, four coated mild steel substrates without post-coating treatment, and three uncoated mild steel were involved in this test. The fatigue test was conducted until the specimen completely fractured or the machine reached 1 million cycles. The number of life cycles was recorded, and the corresponding S-N curves were plotted. Specimens that did not fail up to 1×10^6 cycles were regarded as infinite life. The final data collected from these fatigue tests included the number of cycles to failure for each specimen.

RESULTS AND DISCUSSION

Mass Measurement

Fig 3 illustrates the mass of 30 min coated mild steel substrates subjected to different heat treatment processes. The bar chart represents these mass differences, highlighting that the similar deposition process alone results in the mass differences, with the highest mass recorded by 30 min coating without HT (0.740 g), followed by 30 min coating with annealing (0.710 g). The lowest mass was recorded by 30 min coating with the quenching process, which is 0.690 g. The thermal treatments (annealing and quenching) affect the mass of the samples, possibly through mechanisms such as oxidation, densification, and diffusion. Mugale et al. (2024) discovered that the percentage of coating weight loss increased with longer heat treatment time.

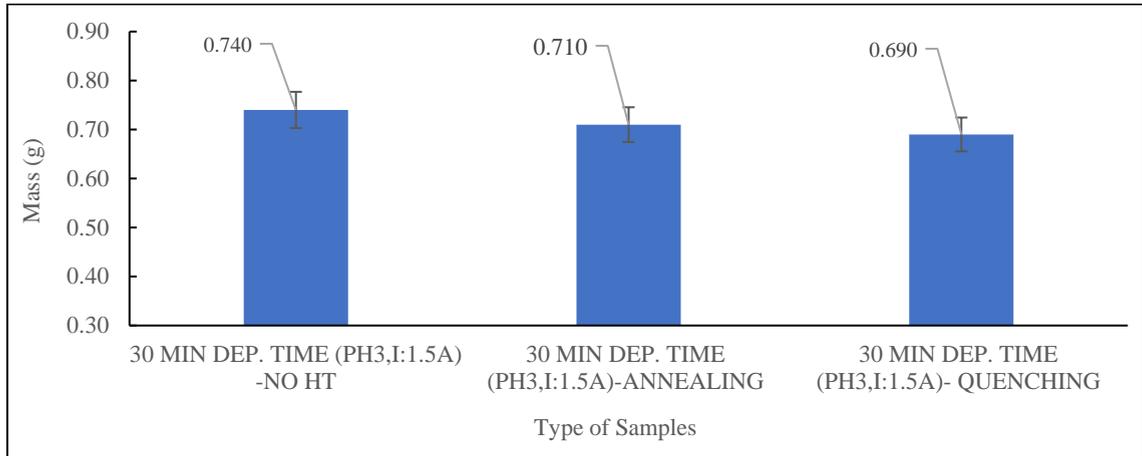


Fig. 3. Mass of 30 min coated mild steel substrates subjected to different post-coating treatment processes.

Fig 4 displays the CoNiFe nanoparticle coating mass at different deposition times. The sample with a 15 min coating possesses the lowest mass (0.35 g), followed by samples with 30 min coating (0.70 g). The highest coating mass was recorded from the 45 min coating period (1.02 g).

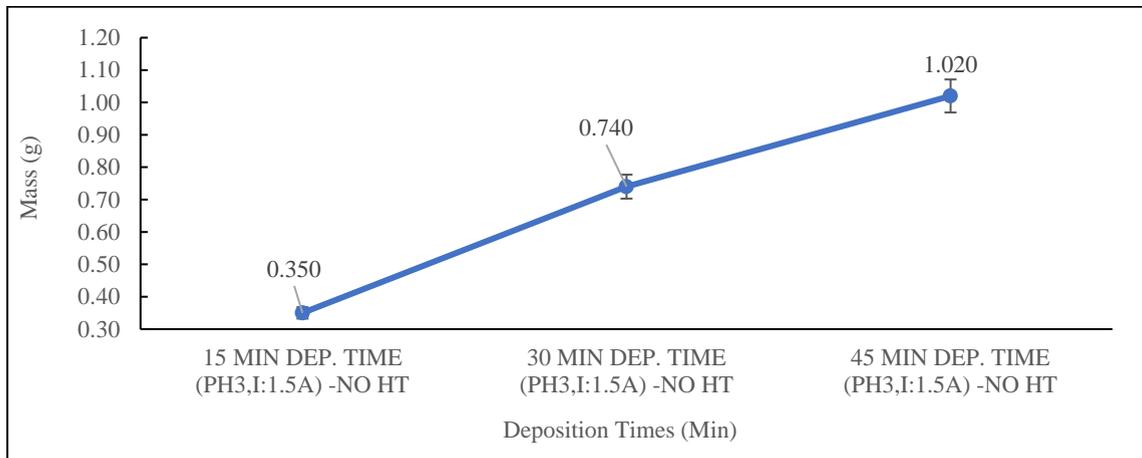


Fig. 4. CoNiFe nanoparticles coating mass at different deposition times.

The graph clearly shows an increasing trend in mass as the deposition time increases. Specifically, the mass of the sample doubles from 0.35 g at 15 min to 0.74 g at 30 min coating. The mass then increases further to 1.02 g at 45 min of coating. This data indicates a direct correlation between deposition time and sample mass. As the deposition time increased, the mass of the sample also increased, suggesting that more cobalt alloy nanoparticles were deposited over longer coating periods. The finding was similarly found by Xiang et al. (2022), who discovered that the deposition weight of PTFE coating on AZ31 magnesium alloy substrate increased with increasing deposition times. Increased coating thickness improves hardness in a variety of ways. Firstly, a thicker coating gives greater surface coverage, lowering the appearance of flaws like pores or microcracks, which can act as stress concentrators and weaken the material. Second, increasing deposition times results in a higher concentration of finer microstructure, which can boost load-bearing

capacity via grain boundary strengthening and dispersion hardening (Deng et al., 2010). Furthermore, a denser coating improves overall resistance to plastic deformation, which increases hardness (Bobzin et al., 2022). These combined effects explain why longer deposition times correlate with higher hardness in the coated sample.

Vickers Hardness

Fig 5 illustrates the Vickers hardness test for CoNiFe nanocrystalline coating at 30 min coating with heat treatment. The uncoated mild steel had a baseline hardness of 132.4 HV, while the mild steel subjected to 30 min coating with no heat treatment, a hardness increased to 217.6 HV. Further treatment with annealing significantly enhanced the hardness up to 266.17 HV. The most substantial increase in hardness is observed on 30 min coated substrates subjected to the quenching process, which resulted in an increase in hardness of 311.62 HV. Heat treatment by the annealing process causes homogenization of the grain and grain coarsening in the coating and substrate, which improves ductility and relieves internal stress (Mayrhofer et al., 2006). However, the material tends to soften compared to the substrate subjected to heat treatment by the quenching process. Quenching process in other ways increases hardness and strength by rapid cooling through the formation of martensite or bainite (Liscic et al., 2014). This progression demonstrates that combining deposition with subsequent heat treatments, especially quenching, greatly improves the material's hardness.

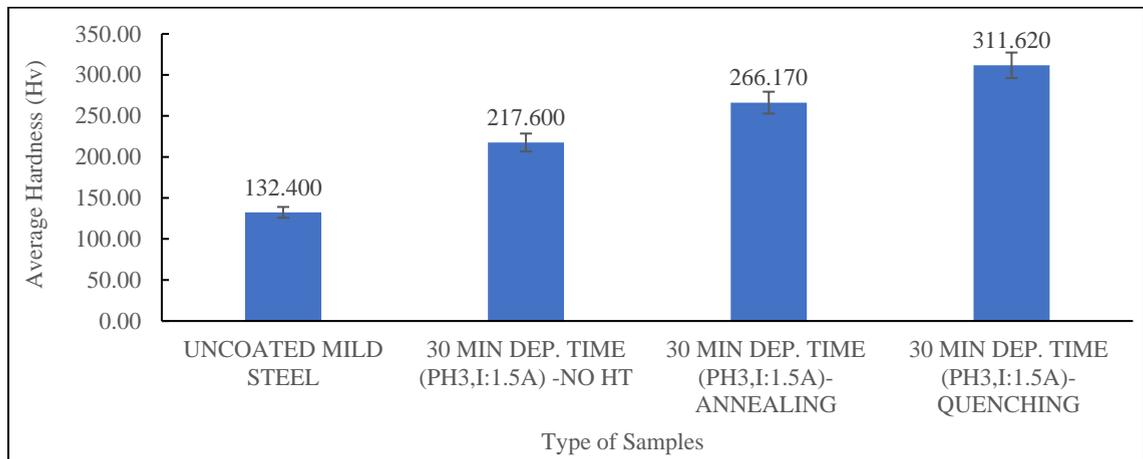


Fig. 5. Vickers hardness test comparison for CoNiFe nanocrystalline coating fixed at 30 min deposition time (heat treatment).

Fig 6 illustrates the Vickers microhardness test for CoNiFe nanoparticles coating at different deposition times. The figure showcases the average hardness for uncoated mild steel and three coated substrates with different deposition times, which are 15 min, 30 min, and 45 min. The baseline hardness value for uncoated mild steel was 132.4 HV. Through the implementation of 15 min coating, the hardness increased to 185.3 HV. The increasing trend can be seen through 30 min (217.6 HV) and 45 min (315.82 HV). The results show a clear trend that as deposition time increases, the hardness of the coated mild steel substrate also increases. As deposition time continues, the increasing hardness indicates a strengthening effect, possibly due to the accumulation of deposited material and subsequent structural changes in the steel. The changes in the phase structure, solid hardening mechanism, impact of porosity, and particle size of the nanoparticles deposit may be contributing factors that affect the steady rise in hardness value from uncoated mild steel to 45 min coating (Hyie et al., 2016). Additionally, a longer deposition period allows for the deposition of more

CoNiFe nanoparticle coating on the mild steel substrate, creating a thicker layer that hardens the coating's surface (Resali et al., 2013).

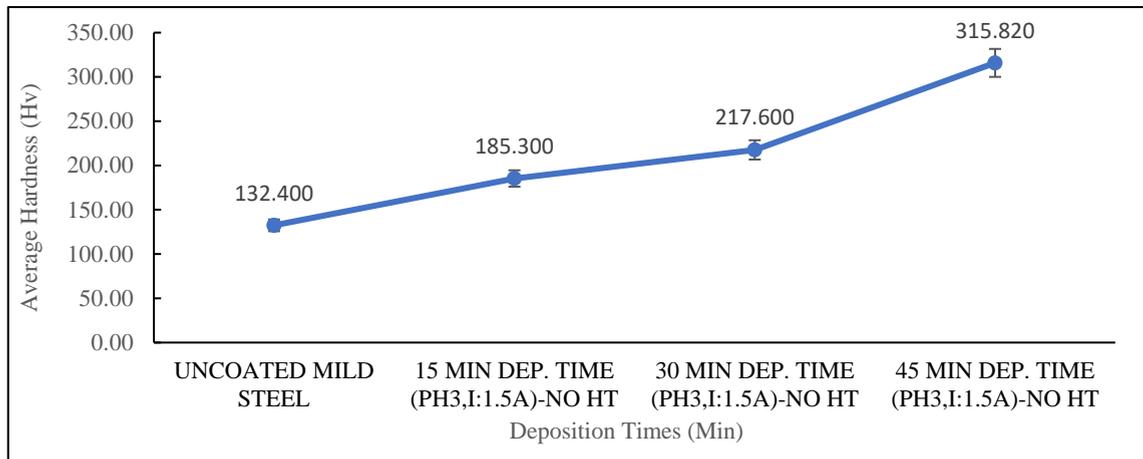


Fig. 6. Vickers microhardness test for CoNiFe nanoparticles coating at different deposition time.

Surface Roughness

Fig 7 illustrates the surface roughness of CoNiFe nanoparticles coated for 30 min with different heat treatment processes. The average roughness values for four different samples: uncoated mild steel, 30 min coating without heat treatment, 30 min coating with annealing, and 30 min of deposition with quenching are shown in Fig 7. The uncoated mild steel had the highest surface roughness, measured at 0.423 μm . This serves as the baseline measurement, indicating the roughness of the untreated material. As the mild steel substrates undergo 30 min of coating at pH 3, current of 1.5 A with no heat treatment, the surface roughness significantly decreases to 0.240 μm .

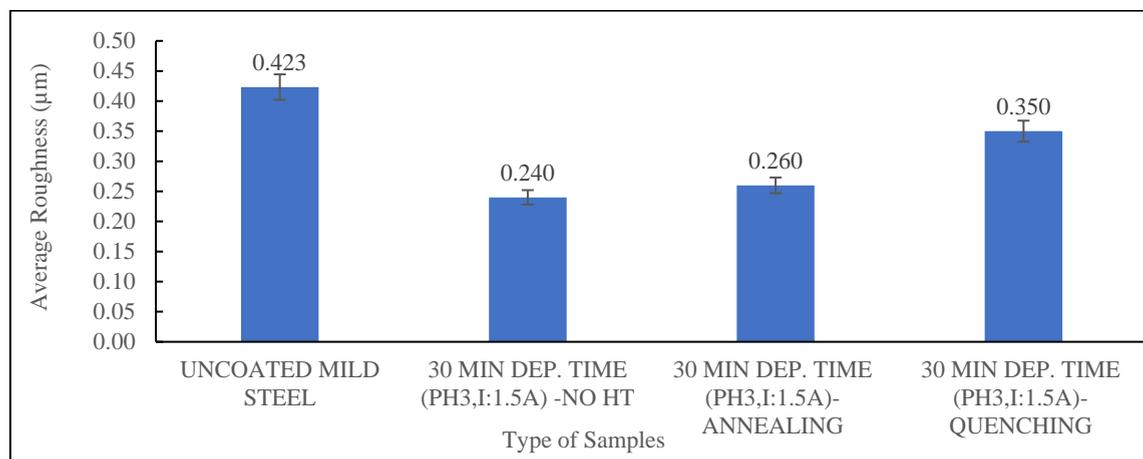


Fig. 7. Surface roughness comparison of CoNiFe nanoparticles coated for 30 min with different heat treatment processes.

Further treatment through the annealing process, following the same deposition conditions, resulted in a slight increase in roughness of $0.26 \mu\text{m}$. Annealing typically involves heating the material and allowing it to cool slowly, which softens the material and hence enhances the ductility and relieves internal stress. A coarse and equiaxed microstructure is usually observed from this treatment. This process may cause minor surface changes, leading to the slight increase in roughness observed. The 30 min coating subjected to quenching further increased the surface roughness to $0.35 \mu\text{m}$. Quenching process involves rapid cooling, which can introduce new surface irregularities due to the sudden temperature change and thermal stresses. This treatment increases the surface roughness compared to 30 min heat treatment (annealing) and 30 min (no heat treatment), but still maintains a lower roughness than the raw mild steel. Quenching substrate normally forms martensite or bainite, depending on the cooling medium (Liscic et al., 2014) which in this study, the authors used water that led to increased hardness.

Fig 8 shows the CoNiFe nanoparticles' surface roughness electrodeposited at different deposition times. Initially, the raw mild steel has a surface roughness of $0.423 \mu\text{m}$, representing the baseline roughness of the untreated material. After 15 minutes of deposition, the surface roughness significantly decreases to $0.2 \mu\text{m}$. As the deposition time increases to 30 minutes, the surface roughness increases slightly to $0.24 \mu\text{m}$. This minor increase suggests that while the deposition continues to improve surface uniformity, it may also introduce slight irregularities or build-up affecting overall roughness. Extending the deposition time to 45 minutes results in a notable increase in surface roughness ($0.39 \mu\text{m}$), approaching the raw mild steel surface roughness value. This increase could be due to the deposition layer becoming uneven over extended periods, possibly from the formation of larger particles or particles stacking onto each other, forming clusters on the surface of mild steel (Hyie et al., 2016). In summary, the data indicates that deposition time significantly affects surface roughness, while additional treatments like annealing and quenching introduce diversity of surface roughness behaviour, which could be observed indirectly through higher surface roughness value.

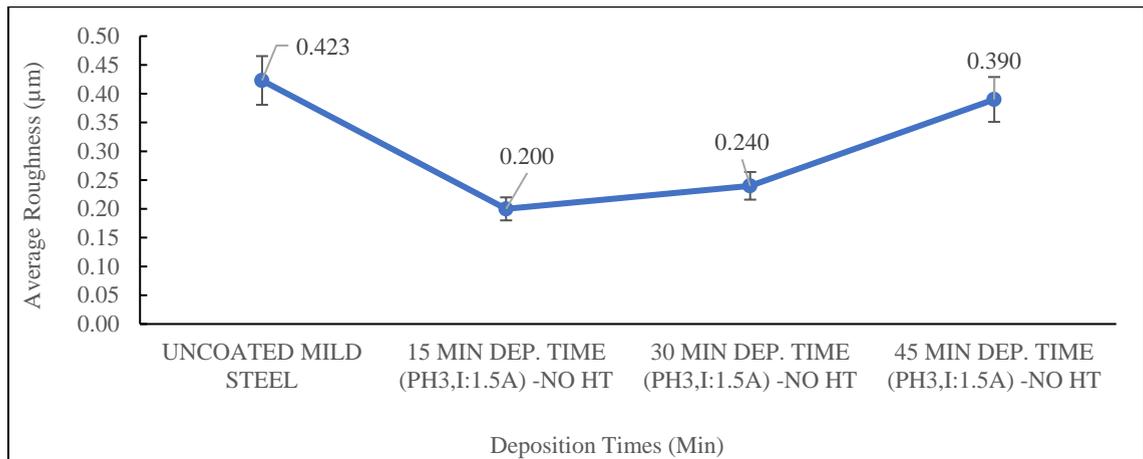


Fig. 8. Comparison of CoNiFe nanoparticles surface roughness electrodeposited at different deposition times.

Fatigue

The number of life cycles to failure of coated and uncoated mild steel substrates is given in Table 4. A total of ten substrates were involved in this test, comprising three uncoated mild steel substrates and seven CoNiFe nanoparticles coated mild steel substrates. Fig 9 shows the maximum stress with respect to cycle time for uncoated mild steel and CoNiFe nanoparticles fatigue test based on different post coating treatment and deposition time. Three different percentages of stress loading were used which were 50%, 80%, and

90% of the maximum ultimate tensile stress (UTS) loading to produce a reference baseline for comparison with CoNiFe nanoparticles coated substrates. The uncoated mild steel, 15 min, 30 min, and 45 min coated mild steel substrates (without additional heat treatment process) were subjected to similar constant of 80% maximum UTS loading. From the perspective of deposition time, the life cycle and maximum stress of 15 min, 30 min, and 45 min coating (without additional heat treatment process) showed an improving pattern of coating compared to uncoated mild steel. The fatigue life of 15 min, 30 min, and 45 min coating (100,000; 200,000 and 270,000 cycles) is higher than uncoated mild steel (1,450 cycles) while the maximum stress recorded for 15 min, 30 min, and 45 min coating (263.0661; 274.6865 and 293.9213 MPa) is lower than uncoated mild steel (315.98 MPa). The CoNiFe nanoparticles deposited on a mild steel substrate act as a strengthening mechanism, bridging surface microcracks. The 'crack-tip bridging' effect facilitated the redistribution of stresses around surface cracks as long as the coating was intact with the steel, delaying crack opening. As fatigue failure is primarily caused by surface cracks rather than embedded cracks, minimizing surface cracks and limiting their propagation would increase the fatigue life of coated specimens when compared to uncoated specimens (Mirghaderi et al., 2021). It was found that the maximum compressive residual stress and the surface compressive residual stress had a direct correlation with the substrates fatigue life (Smith et al., 2007). The longer substrates fatigue life was consistently at least as good as that of the uncoated mild steel, even though the higher fatigue life compensates with lower maximum stress. By maximizing the percentage of loading to 90%, it could be observed that the best coated substrates (30 min heat treatment-annealing; 80,000 life cycle; 293.45 MPa) surpassed the life cycle value of similar loading percentage on uncoated mild steel (1,200 life cycle; 293.72 MPa) by a whopping 166.67% with negligible difference in maximum stress value. However, the trend for 80% loading observed coated substrate (30 min heat treatment-quenching; 1,000,000 life cycle; 262.75MPa) surpasses the result of similar loading percentage on uncoated mild steel (1,450 life cycle; 315.98 MPa) by a superior duration of life cycle by 689.66%.

Table 4. Comparison of uncoated mild steel and CoNiFe nanoparticles fatigue test based on different post-coating treatment and deposition time

Sample type	Loading, %	Life cycle	Maximum stress (MPa)
Uncoated mild steel	80	1,450	315.98
Uncoated mild steel	90	1,200	293.72
Uncoated mild steel	50	1,000,000	183.95
30 min (no heat treatment)	80	90,000	269.41
30 min heat treatment (annealing)	80	200,000	270.87
30 min heat treatment (quenching)	80	1,000,000	262.75
30 min heat treatment (annealing)	90	80,000	293.45
15 min (no heat treatment)	80	100,000	263.0661
30 min (no heat treatment)	80	200,000	274.6865
45 min (no heat treatment)	80	270,000	293.9213

For comparison with constant 30 min coating (different post coating treatment), all the 30 min coating substrates (different post coating treatment) were subjected to a similar constant of 80% maximum UTS loading. It can be observed that the introduction of heat treatment such as annealing and quenching elevates the fatigue life of the coated substrates. The lowest fatigue life was recorded by 30 min coated substrate (no heat treatment) at 90,000 life cycles (max stress: 269.41 MPa) followed by 30 min coated substrate (heat treatment-annealing) which recorded 200,000 life cycles (max stress: 270.87 MPa). The highest life cycle is obtained by 30 min coated substrate (heat treatment-quenching) with 1,000,000 cycles (max stress: 262.75 MPa). Stress amplitude at 1,000,000 cycles was considered as the fatigue limit (machine maximum limit-infinite life). Annealing processes usually soften the material which relates to higher fatigue. Not just that, the coarsening and coalescence of the substructure might be obtained when the annealing process takes place (Mayrhofer et al., 2006). However, quenching usually makes material harder by introducing localized stress and more brittle than annealing substrates. Quenching method normally forms a martensite or bainite, depending on the cooling medium (Liscic et al., 2014). The finding by Liscic et al. (2014) is correlated with

this study, as listed in Table 4. It can be observed that 30 min Heat Treatment (Quenching) and 30 min Heat Treatment (Annealing) coated substrates recorded a life cycle of 1,000,000 and 200,000 life cycles, respectively. Annealing substrates shows a high tendency of fatigue, while quenching substrates can withstand a high life cycle provided the load ratio is constant at 80% of UTS. As the quenching substrates might possess brittle characteristics, the maximum stress of the quenching substrate over the annealing substrate would be less, which in this case was recorded as both 262.75MPa and 270.87 MPa, respectively. The presence of CoNiFe nanoparticles potentially enhanced the material's ability to withstand cyclic loading over an extended period. Comparison of uncoated mild steel and 30 min heat treatment (annealing) subjected under higher UTS loading (90%), recorded a higher life cycle (80,000 cycle) and maximum stress (293.45 MPa) compared to uncoated mild steel subjected to similar percentage of UTS loading (life cycle: 1,200 and maximum stress: 293.73 MPa). This proves that the introduction of the heat treatment process after coating can handle higher stress while also giving a much longer life cycle. From a previous study, it also stated that coated and treated samples will improve the mechanical properties of the material, such as increasing fatigue strength (Mirghaderi et al., 2021; Tsai et al., 2019; Komotori et al., 2001).

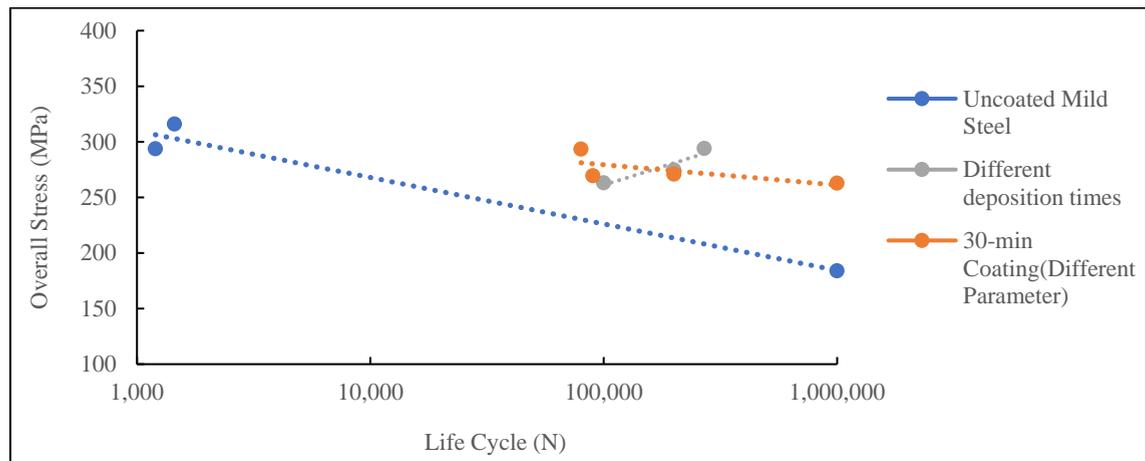


Fig. 9. Comparison of overall stress vs cycle time graph uncoated mild steel and CoNiFe nanoparticles fatigue test based on different post coating treatment and deposition times.

CONCLUSION

In this study, the introduction of CoNiFe nanoparticle coating together with the post-coating process using heat treatment, such as annealing and quenching, was investigated. It can be concluded that CoNiFe nanoparticles coating itself significantly enhances the properties of mild steel substrates. The combination notably increases the hardness, which demonstrates that longer deposition time further improves hardness. Prolonged deposition time and additional treatments such as annealing and quenching resulted in an increase in surface roughness. Therefore, managing deposition time is crucial for maintaining the desired surface characteristics. Throughout fatigue testing, it can be seen that coated mild steel substrates exhibit higher fatigue life, indicating enhanced durability under cyclic loading. The data shows an inverse relationship between overall stress and fatigue life, where higher stresses lead to shorter life cycles. Overall, the study shows that electrodeposited coatings combined with heat treatments effectively improve the hardness, surface roughness, and fatigue resistance of mild steel substrates, making it suitable for applications requiring enhanced mechanical applications.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS' CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: **study conception and design:** M.Z Zabri, N.R.N Masdek; **data collection:** M.Z Zabri, M.A.M Syehab; **analysis and interpretation of results:** M.Z Zabri, N.R.N Masdek; **draft manuscript preparation:** M.Z Zabri, M.A.M Syehab. All authors reviewed the results and approved the final version of the manuscript.

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