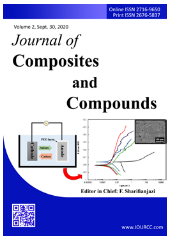

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Ni-Cu matrix composite reinforced with CNTs: preparation, characterization, wear and corrosion behavior, inhibitory effects

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ABSTRACT

St37 steel has been used in various industries due to its abundance and low cost. However, the high corrosion rate of steel in acidic environments is one of the limiting factors for its application. In this study, Ni-Cu composite coating reinforced with CNTs was applied on the st37 steel substrate. The extract of the Sarang Semut plant was added to the coating as inhibitory particles and the electrochemical behavior of the coating was investigated. The X-ray diffraction test was performed for phase analysis. Hardness, wear, and dynamic potential polarization tests were performed. Results showed that the presence of CNT particles improved the hardness, tribological performance, and electrochemical behavior of the coating. Also, the presence of Sarang Semut particles acted as a barrier and protected the surface of st37 steel from corrosion. It should be noted that these particles affected the kinetics and thermodynamics of corrosion reactions and were not involved in the reactions.

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1. Introduction

St37 steel is structural steel available in various forms. This type of steel is inexpensive and can be used in different applications including plant fabrication, piping, etc. Carbon is the main alloying element in the steel and there are some trace impurities as well. The hardness and strength of the steel are enhanced with the increment of the carbon content [1-4]. As steel and its alloys are extensively utilized in industrial components, their protection against acid electrolytes and their corrosive effect is necessary [5-7]. Common protection methods are alloying steel with inhibiting elements, using corrosion inhibitors, cathodic and anodic protection, and protective coatings [8, 9]. In various applications like the automotive and aerospace industry, composite coatings prepared via co-deposition of small reinforcement particles into a metal matrix are extensively used [10, 11]. It has been reported that the composite coatings could enhance microhardness, high-temperature stability, wear resistance, and corrosion resistance of metal and alloy. There are several factors that should be considered in selecting these coatings such as technical constraints, environmental pollution, costs, simplicity of the coating process [12-15].

In the marine environment, Ni-Cu alloys, particularly alloy that contain about 70%, Ni shows high corrosion resistance. Apart from corrosion resistance, solderability, ductility, and malleability are other attractive features. Cu and Ni have the standard reduction potentials of +0.34 V and 0.25 V vs. a normal electrode of hydrogen (NHE), respectively, which are disparate to some extent. A normal co-deposition of Ni and Cu is the plating process of Ni-Cu alloy. The incorporation of Cu in Ni strengthens it owing to the formation of a solid solution that enhances wear and corrosion resistance [16].

Besides the selection of the coating method, optimization of the electroplating method and the alloying system is also necessary. Some applicable techniques include electrodeposition, physical vapor deposition, chemical vapor deposition, plasma spraying, and thermal spraying. For the preparation of Ni coatings, electrodeposition has been reported to be a proper technique [17]. The reduction of the corrosion rate could occur in the alloy by adding metallic (e.g., Cr, or Mo) or nonmetallic (e.g., P, N or Si) inhibiting components, which forms adsorbed intermediate products on the alloy surface and blocks the active areas [8].

Since their emergence, carbon nanotubes (CNTs) have been increasingly used for the fabrication of advanced composites as the ideal reinforcing agent owing to their rewarding mechanical properties such as fracture strain sustaining capability, light weight, large elastic strain, and high elastic modulus [18-20]. However, CNT-containing composite applications are accompanied by the high complexity because several factors such as the nanotube/matrix bonding, nanotubes dispersion, reinforcement aspect ratio, surface reactivity, and densification of bulk composites should be controlled [8, 21-23]. In this research, the Ni-Cu/CNT composite coating is deposited on the st37 substrate, and its mechanical and corrosion behaviors as well as the inhibitory property of CNT on the coating surface were evaluated.

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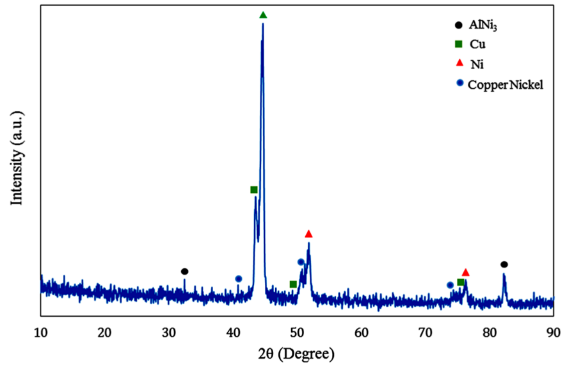

Fig. 1. XRD pattern for composite coated specimen.

2. Materials and method

2.1. Substrate preparation

The substrate used in this research for coating is St37 steel, and its chemical composition is shown in Table 1. The samples with 20×20×1 mm^3 dimensions were sanded using silicon carbide sandpaper (No. 60 to 3000) and then polished with alumina solution to reach an appropriate surface. Then the samples were washed with acetone and distilled water in order to remove the impurities from the surface and acid-washed by sulfuric acid (10 vol. %).

2.2. Preparation of electrolyte and coating

To prepare the electroplating bath, 100 g/L of $NiSO_4.6H_2O$, 25 g/L of $CuSO_4.5H_2O$, 32 g/L of H_3BO_3, 25, 100, and 500 mg/L of CNT, 0.30 g/L of sodium dodecyl sulphate (SDS) (German company Merck) were used. The complexing agent used in this study is $Na_3C_6H_5O_7$ (65 g/L). The conditions for applying the coating are listed in Table 2. The inhibitor used in this study is Sarang Semut, which has been studied by Shahab et al [24]. They prove that the extract of this plant has inhibitory properties in 3.5 wt. % sodium chloride solution. The extract was prepared from this plant (according to the research of Shahab et al.) and was completely pulverized after the production. In order to observe the effect of the presence of this extract on the composite coatings reinforced with CNT particles, 500 mg/L of this powder was mixed with 200 mg/L of reinforcing CNT particles, and then the mixture was added to the electroplating bath as the composite agent. The coating deposited in this bath was subjected to electrochemical tests.

The anode used in this study is a pure nickel sheet (99.99%) with 10×10×50 mm^3 dimensions. The anode and cathode were immersed vertically in a 400 cc of bath with a distance of about 5 cm between the anode and cathode and connected to the power supply by suitable electrical connections.

2.3. Characterization of coatings

Phase analysis of the coating was carried out by X-ray diffraction (XRD) using Philips Xpert pro type with Cu kα_1, 2θ range of 20-100°, step size of 0.03 at a voltage of 20 kV. Tribological behavior of the samples using the pin on disc was studied by BONG SHIN model obu-50 in humidity conditions of 35% and at a temperature of 31.5 ° C. The pin rotation speed was 0.125 m/s and wear distance was 50 m and the test was performed without lubricant. The conversion factor in these tests was 26.32 and the applied force was 10 N. The pin was made of hardened steel with 50 HRC hardness.

Table 1.
Chemical composition of steel used as substrate

Element	P	Mn	Si	S	C	Fe
wt. %	0.05	0.32	0.33	0.05	0.18	99.03

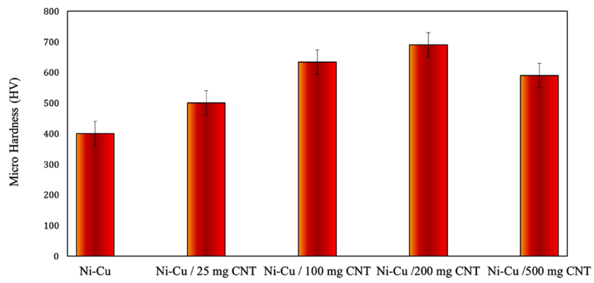

Fig. 2. Microhardness results of the applied coatings.

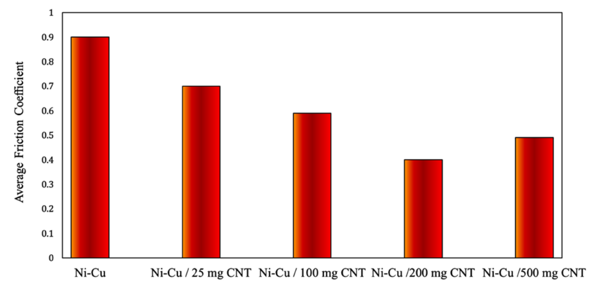

Fig. 3. Friction coefficient changes for coated specimens.

Potentiodynamic polarization (PDP) test was used to investigate the corrosion behavior of the samples. The samples were immersed in 3.5 wt. % sodium chloride for 1 hour to balance the chemical potential. Electrochemical experiments were carried out in a three-electrode-type cell with different compartments for the reference electrode (SCE). The counter electrode was a platinum plate, while the working electrode was St37 steel. Before each experiment, the working electrode was immersed in the test solution for 2 h at 25 °C to attain a stable open circuit potential (OCP).

The micro-hardness test was performed using the German Buhler machine, MMT-V model with Vickers submersible according to ASTM-E 384 standard under a load of 10 g for 10 seconds. The tests were randomly performed and finally, the average test results were presented.

Table 2.
Parameters of coating application

Parameter	Value
Temperature (°C)	35
Current density (A/dm^2)	2
Steering rate (rpm)	350
pH	4-5

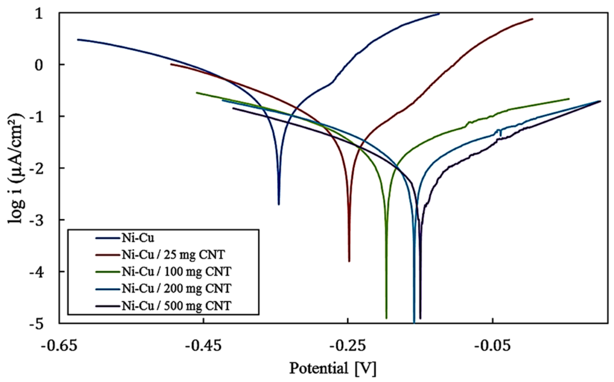

Fig. 4. PDP test results in 3.5% NaCl electrolyte.

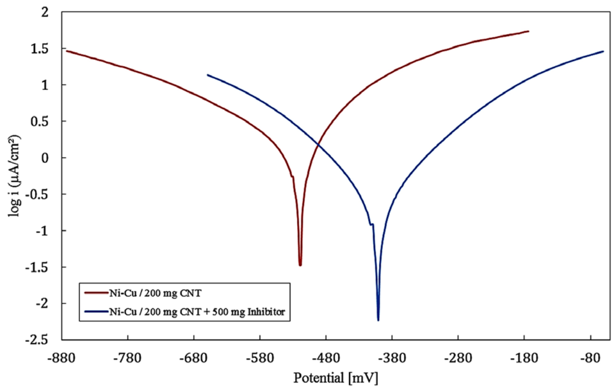

Fig. 5. PDP test results in 3.5% NaCl electrolyte for distorted specimens containing no inhibitor.

3. Results and discussion

3.1. XRD results

Fig. 1 indicates the XRD results of Ni-Cu/CNT alloy coatings. Fig. 1 shows that the Ni and Cu peak is present only in the coating. According to the figure, it is clear that nickel and copper together formed a solid solution leading to the formation of the nickel-copper alloy phase, and finally, the peak of this solid solution is observed, which has been proven in other studies as well [25, 26]. The conducted studies have shown that the presence of Cu atoms in the structure of Ni leads to a decrease in the lattice parameter and ultimately shifts 2θ to a lower value [27]. The figure clarifies that CNT peaks are seen in the X-ray pattern as well. This shows that CNT composite particles exist in the coating structure. Thus, the existence of CNT peaks in X-ray diffraction patterns shows that the fabrication of Ni-Cu/CNT composite coatings is possible using the electrodeposition method. Some studies show that the presence of composite particles like CNT or the presence of their alloying elements and cations like Cu^{2+} has led to changes in the energy of the growing plates. As the structure of the applied coating is related to the energy of the growing plates, the presence of CNT and Cu^{2+} particles leads to changes in the texture and structure of the specimens. It has been indicated that the changes in the texture of the specimens led to changes in the electrochemical and tribological behavior of the specimens [28-30].

According to Fig. 1, it is shown that peaks related to carbon nanoparticles are not seen, which may be due to the high density of peaks related to Ni or Cu. Another factor for the absence of peaks related to CNTs is their low concentration. This has been confirmed in the case of C_3N_4 carbon nitrite nanoparticles too [31].

3.2. Mechanical behavior of the specimens

The micro-hardness test was used to study the mechanical behavior of the specimens. Fig. 2 shows the micro-hardness results of the specimens. The hardness test was conducted to evaluate the micro-hardness of coatings applied to the cross-section of the specimens. Fig. 2 shows that composite specimens have a higher hardness compared with the Ni-Cu specimens. According to studies, it has been found that the hardness of the coating increases with the presence of Cu in the coating. The reason for the increase in hardness is related to the presence of Cu atoms in the correction of orientation and growth of coating grains [17, 30]. This issue can be seen in this study too. Fig. 2 shows that the hardness of the specimens increased with the presence of the secondary phase of CNT in the coating and the creation of Ni-Cu/CNT composite coating. The increase in hardness due to the presence of the secondary phase can be analyzed by several mechanisms. Texture change, change in grain boundary energy in the presence of secondary phase (Hall–Petch equation), dispersion strengthening (Orwan theory) are the causes that explain the increase in hardness in the presence of secondary particles [30, 32-34]. According to Orowan's theory, it is clear that the presence of CNT particles locks dislocations and hence prevents them from moving and ultimately leads to an increase in the hardness of the specimens.

Carbon nanotubes are one of the hardest materials in nature that have high strength and can withstand up to 55 GPa pressure conditions without degrading the tubular shape. The hardness of single-walled nanotubes can be up to 62 to 150 GPa. According to some studies, CNTs showed a much higher hardness compared to SiC in the nickel-phosphorus coating, which subsequently improved the abrasion properties.

According to Fig. 2, it is clear that the presence of nanotubes increases the hardness of the coatings. The presence of 25 mg/L of nanotubes increased the hardness from 400 to approximately 500 Vickers. The further increase in the concentration of nanotubes in the bath and the possibility of greater participation of nanotubes in the coating increase the hardness and reinforcing particles with uniform distribution inside the coating, thus reduce the role of the ground phase in the final hardness and load-bearing capacity by more particles. Therefore, the role of the matrix in the final hardness of the coating is reduced. Moreover, it is clear that by the increase in the nanotube concentration from 200 mg/L to 500 mg/L, the hardness of the specimens decreased. This can be because of the agglomeration of carbon nanoparticles. Carbon nanotubes agglomerate in 500 mg/L concentration and do not properly distribute, leading to the reduction of engineering properties of the specimens, including hardness. In three-component Ni-Cu-P-nano graphite composite coating, where lower concentrations of copper were used in the soluble compounds, the hardness increased due to the strengthening effect of the reinforcement particles. However, the hardness reduced in the solution with more copper compounds.

3.3. Tribological behavior of coatings

Pin-on-disk wear testing was used to study the tribological behavior of the specimens. The changes in the coefficient of friction of the specimens are shown in Fig. 3. According to this figure, it is clear that the average friction coefficients of composite specimens are lower than that of the Ni-Cu specimens. This shows an improvement in the tribological behavior of the specimens in the presence of the composite particle. The presence of carbon nanotubes as reinforcements has reduced the friction coefficient, which can be attributed to the atomic structure and shape of their tubes, leading to the property of self-lubrication [35, 36]. According to some studies, CNT particles increase coating hardness and strength (Hall–Petch and Orwan theory) [37], so they prevent the plastic deformation of the specimens during the wear test. During the abrasion process, the loss of material on the surface makes one end of the nanotubes placed vertically in the coating to be removed from the coating and due to the pressure of the pin, it becomes curved and the pin starts moving on its walls. Thus, this movement reduces the coefficient of friction [38]. Carbon nanotubes are made of concentric layers or walls of carbon tubes and the bonding of the layers is of the van der Waals type so they can easily slide over each other [39]. Dye et al. reported that nanotubes that detach from the surface act like a microbearing. CNTs can rotate, separate slippery surfaces, prevent strong contact between the two surfaces; thus, they reduce the friction coefficient, and increase wear resistance [31, 40].

It is clear that in the wear test, with an increase in the concentration of CNT to 500 mg/L, the coefficient of friction increased compared to the specimen of 200 mg/L, which can be analyzed by CNT agglomeration in the coating as well.

Among composites, carbon-reinforced composites have better abrasion behavior compared to ceramic-reinforced composites like Al_2O_3 or SiC, which is due to the lubricating nature of the carbon materials that make them natural reinforcement for lubricating composites [41].

3.4. Electrochemical behavior of coatings

3.4.1. Examining the electrochemical behavior of composite coating

The potentiodynamic polarization (PDP) test in 3.5 wt. % sodium chloride was used to examine the electrochemical behavior of the coatings. Fig. 4 shows the results of the PDP test. According to this figure, it is clear that the application of composite coatings caused a change in the electrochemical behavior of the specimens. This can be recognized by changes in the Tafel branches and the potential of the specimens. According to Fig. 4 and Table 3, it is clear that the presence of reinforcement particles in any amount has reduced the corrosion flow rate of the specimens [33, 42-46]. The reduction in the corrosion rate of composite specimens compared to the Ni-Cu alloy specimens can be due to a higher density of composite specimens. Due to the low ion radius of Cl^-, it is clear that increasing the density of the coating and reducing the micropores in the coating leads to the enhancement in the electrochemical behavior of the specimens. This is because increasing the density of the coating leads to a decrease in the penetration rate of this ion and ultimately the corrosion rate is reduced [47]. This has been proven in other studies [32, 33]. The improvement in the electrochemical behavior of the specimens due to the presence of CNT particles, in addition to the above, is related to the coating of metal sites by these particles in such a way that these particles reduce the charge transfer by creating electrical resistance between the electrolyte and the substrate. Ultimately, it enhances the electrochemical behavior of the specimens [34]. By studying the potential of the specimens, it is seen that the composite specimens have a nobler potential compared to the Ni-Cu specimen. The reason why the potential of composite specimens is nobler than Ni-Cu specimens is the presence of CNT particles. CNT shifts the potential of the specimens to more positive and noble values. This decreases the thermodynamic tendency of the specimens to perform corrosion reactions [48]. It is further clear that the anodic and cathodic slopes will undergo drastic changes if reinforcement particles are added. In other words, there is no parallel line between the branches of Ni-Cu diagrams and the composite specimens. This shows that the application of composite particles affected the mechanism of anodic and cathodic reactions [49].

Table 3.
Results extracted from PDP test by Tafel method

Sample	i_{corr} ($\mu A.cm^{-2}$)	E_{corr} (V)	β_a (mv/decade)	β_c (mv/decade)
Ni-Cu	3.16	-0.346	99	-84
Ni-Cu / 25 mg CNT	0.312	-0.248	96	-83
Ni-Cu / 100 mg CNT	0.031	-0.197	90	-79
Ni-Cu / 200 mg CNT	0.019	-0.158	91	-80
Ni-Cu / 500 mg CNT	0.012	-0.150	89	-81

3.4.2. Examining the electrochemical behavior of coating in the presence of inhibitor

It is clear that the presence of inhibitory particles in corrosion systems will reduce the corrosion rate. Thus, the use of inhibitors in the systems involved in corrosion reactions is one of the ways to prevent corrosion reactions. Inhibitors are used in corrosive soluble forms in corrosion protection systems. In this study, inhibitory particles were used as particles in the coating. Thus, the coating has inhibitory particles and if there is a scratch or scar in the coating, the inhibitor will be removed from the coating and it will protect the part of the base metal that does not have a coating by using inhibitory mechanisms. To this end, 500 mg/L of the inhibitory particles were mixed with 200 mg/L of CNT, and then the resulting powder was introduced into the plating bath as reinforcement particles. Ultimately, after coating, a small 5 mm scratch was applied to the coating, the specimen reinforced with inhibitory particles, and CNT was tested for PDP. Another specimen that does not contain inhibitory particles (Ni-Cu/200 mg CNT) was scratched about 5 mm and then subjected to PDP under the same conditions to compare the electrochemical behavior of this coating with coatings without inhibitory particles. Fig. 5 and Table 4 show the PDP test results for two composite specimens with and without the inhibitor.

Equation 1 was used to measure the inhibition percentage [50]:

$$\% IE = \frac{i_{Corr} - i_{Corr(Inhibitor)}}{i_{Corr}} \times 100 \tag{1}$$

In this equation, i_{Corr} is the corrosion current density in the absence of the inhibitor and $i_{Corr(Inhibitor)}$ is the corrosion current density in the presence of the inhibitor.

Fig. 5 and Table 4 clearly show that, firstly, by scratching the specimens, the corrosion rate of the specimens increases compared to healthy specimens. The presence of scratches in the coating facilitates the attack of ions like Cl^- and finally brings about an increase in the formation rate of the base metal chloride, which increases the corrosion rate of the specimens too. To prevent this, the study used inhibitory particles inside the coating. In other words, a coating is prepared by the incorporation of inhibitory particles. In case of damage to the coating and stripping of the base metal, the inhibitory particles enter the environment from the scratched area and protect the stripped metal site. The protection of the base metal is shown in Fig. 5 and Table 4. According to Fig. 5 and Table 4, it is clear that the corrosion rate in the inhibitor-containing specimen is lower than the specimen without the inhibitor, which is a reason for the inhibition by the particles applied in the coating. It is clearly seen that the potential of the scratched specimen shifts to more noble values with the presence of a scratched inhibitor. This shows that the thermo-

Table 4.
The results of Tafel PDP test for distorted specimens without and with inhibitor

Sample	i_{corr} ($\mu A.cm^{-2}$)	E_{corr} (V)	β_a (mv/decade)	β_c (mv/decade)	I.E (%)
Ni-Cu / 200 mg CNT	10.32	-0.518	95	-87	-
Ni-Cu / 200 mg CNT + 500 mg Inhibitor	5.011	-0.248	96	-83	51

dynamic inhibitory particles affect the corrosion reactions at the site of the bare metal and reduce corrosion reactions. As the corrosion rate decreases, it is clear that the kinetics of corrosion reactions are affected by the presence of inhibitors at the bare metal site, and in the presence of inhibitors, the kinetics of electron transfer is slowed down. Given the parallel state of Tafel branches, it is clear that the presence of inhibitory particles in the coatings has only affected the kinetics and thermodynamics of corrosion reactions and it was not able to change the mechanism of corrosion reactions in the present conditions [51].

4. Conclusions

In this research, the Ni-Cu alloy coating containing carbon nanotubes was produced. Then, the particles of the Sarang Semut plant were added to the coating as inhibitory particles and the electrochemical behavior of the coatings was investigated in the case of damaged coating in the presence of inhibitory particles. The results of the studies are as follows:

- In the Ni-Cu coating, CNT particles can be present as a composite agent.
- The presence of composite particles up to 200 mg/L improved the hardness of the samples compared to the pure Ni-Cu sample.
- Tribological behavior of the composite samples was more favorable than pure Ni-Cu samples and in the presence of composite particles up to 200 mg/L, the coefficient of friction of the samples decreased.
- By increasing the concentration of composite particles from 200 mg/L, the composite particles agglomerated and decreased the engineering properties of the samples such as mechanical and tribological properties.
- The presence of carbon nanotubes in the coatings led to the improvement of the electrochemical behavior of the samples due to the blockage of micropores, making the potential of the coatings nobler and creating insulation between the metal substrate and the electrolyte.
- The presence of Sarang Semut particles in addition to carbon nanotubes improved the electrochemical behavior of the coatings.
- In the coatings reinforced with CNT and Sarang Semut, in case of scratches in the coating, Sarang Semut particles act as an inhibiting agent and protect the bare surface of the metal from corrosion.
- Sarang Semut as an inhibitor is not able to change the mechanism of corrosion reactions and only affects the kinetics and thermodynamics of corrosion reactions.

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Conflict of Interest

All authors declare no conflicts of interest in this paper.

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