Investigation of Wear Resistance and Corrosion of Ni-PTFE Composite Coatings Prepared by Electrodeposition Method

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Editor's note: Composite coatings are recognized for their effectiveness in providing corrosion protection and wear resistance. An investigation was conducted by Wetwet on the impact of varying concentrations of polytetrafluoroethylene (PTFE) particles on the corrosion inhibition and wear resistance of nickel-based coatings applied to a St37 steel substrate. The results indicate that achieving an optimal concentration of PTFE through the electrodeposition coating process significantly enhances the wear resistance and corrosion inhibition performance of nickel-based coatings. This makes the final coating ideal for developing metal-based infrastructures that require corrosion resistance.

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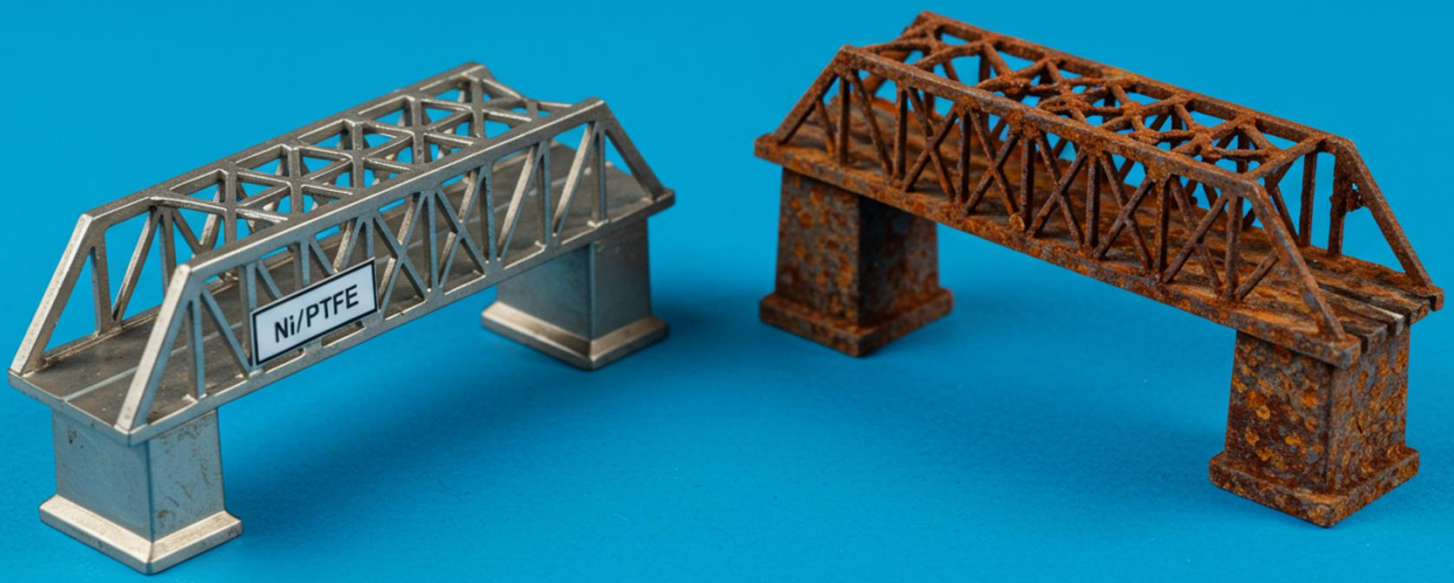

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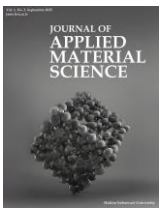

Original Research

Investigation of Wear Resistance and Corrosion of Ni-PTFE Composite Coatings Prepared by Electrodeposition Method

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Abstract

The results of various research activities have shown that coating is one of the effective ways to increase the corrosion resistance and wear resistance of metallic substrates. Additionally, composite coatings using nanoparticles can also provide further protection to the substrate. In this study, Ni-PTFE coatings were prepared using the electrodeposition process with polytetrafluoroethylene (PTFE) particles at concentrations of 10, 20, or 30 g/L. Their corrosion and wear properties were investigated and compared to those of Ni-P coatings. Using scanning electron microscopy and energy dispersive spectroscopy, the surface morphology and elemental composition of the coatings were analyzed, and finally, by using open circuit potential techniques, electrochemical impedance spectroscopy, and Tafel polarization techniques, the corrosion resistance of the resulting coatings in 3.5 wt.% NaCl solution was evaluated. Microhardness and pin-on-disk tests were also utilized to investigate the effect of PTFE concentration on the tribological properties of the coatings. The results of scanning electron microscopy and energy dispersive spectroscopy studies confirmed the formation of nanocomposites. Electrochemical studies indicated that Ni-PTFE coatings at a concentration of 20 g/L PTFE exhibited the highest electrochemical corrosion resistance. Microhardness decreased as the amount of PTFE particles in the coating increased, reaching its lowest value. By using the wear test, the lowest coefficient of friction was obtained in composite coatings with a concentration of 20 g/L, which shows the applicability of PTFE particles as a solid lubricant in Ni-P coatings.

Keywords: Electrodeposition; Composite coatings; PTFE; Wear; Corrosion; EIS.

1. Introduction

Applying various types of protective coatings is one way to reduce costs associated with corrosion and wear in different industries [1]. Among the various coating methods, such as electroless plating, phosphating, and electrodeposition, nickel coatings have been widely used in the industry [2, 3]. Since 1843, when the first chemical compound of the nickel plating bath was introduced on an industrial scale from nickel-ammonium sulfate solution, until today, many researches have been proposed on improving the properties of these coatings, because these coatings have wide applications in various industries such as aerospace, automotive and chemical industries such as oil and gas [3].

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One of the most important solutions to improve properties is to add secondary elements such as iron [4] or phosphorus [4, 5] or phosphorus [5] or composite the coating by introducing micron-sized particles or nano-like SiC to the field of nickel coating [6, 7]. Adding 10 g/L of graphene oxide to the nickel plating bath has been reported to reduce the corrosion rate by one-fifth [8]. The simultaneous presence of iron and TiC ceramic particles in the nickel coating structure results in a corrosion current density of 0.79 μA/cm² [9].

Several studies have demonstrated that wear and friction are intricate processes influenced by the surface properties of the two sliding objects, the tribological conditions, and their surrounding environment. Many dynamic systems are lubricated with various lubricating fluids. However, one disadvantage of these lubricating liquids is their inability to perform at high temperatures or under vacuum conditions, as they generally lose effectiveness in such environments. Most research has focused on another materials category that reduces the friction coefficient, known as solid lubricants [10, 11].

In recent years, many studies have explored the use of particles such as WS_2 and MoS_2 as solid lubricants in coatings [12, 13]. It has been observed that when solid lubricants are applied to the surfaces of parts operating under slippery conditions, they create a layer between the two surfaces that are in contact. This layer prevents the surfaces from making contact and welding together due to their differing characteristics. Some solid materials exhibit low shear strength, and when they are placed on a slippery surface, they significantly decrease wear and friction. This indicates that the use of solid lubricants, such as protective coatings, is necessary [13, 14]. For example, research results have demonstrated that in the presence of WS_2, the friction coefficient of Ni-P coatings obtained by the electrodeposition method decreases from 0.5 to 0.17 [12]. The presence of MoS_2 in these coatings also reduces the friction coefficient from 0.45 to 0.05 [13]. Another type of solid lubricant is PTFE, whose effect on reducing the friction coefficient of various coatings such as Ni-W has been studied [15]. However, the effect of the presence of this material in the field of Ni-P coatings has not been comprehensively studied. The optimal concentration of PTFE for maximum corrosion resistance, wear resistance, and hardness has not yet been investigated.

In this research, different concentrations of PTFE were deposited on the steel base in Ni-P PTFE electrodeposition baths, and the general characteristics of the coatings, such as chemical composition, participation rate of PTFE in the coating, surface morphology, and hardness of the coating, were evaluated. The effect of the participation percentage of PTFE in the coating on the corrosion behavior was studied using the Tafel polarization test and electrochemical impedance spectroscopy (EIS) in an environment containing 3 to 5% by weight of sodium chloride. Finally, the tribological behavior of the coatings was investigated using the pin test on the disc, and the effect of the participation percentage of PTFE particles in the coating on the friction coefficient was determined. In this study, we utilized soft lubricant particles, specifically PTFE particles, in combination with electroless nickel coating to enhance the friction properties of the surface. The PTFE particles are valued for their chemical stability and unique physical and mechanical properties, which can simultaneously improve the corrosion resistance and tribological behavior of composites.

2. Experimental

2.1. Materials

Sodium phosphate metahydrate and sodium carbonate were purchased from Sigma-Aldrich. Hydrogen peroxide and sulfuric acid were purchased from Merck (Germany). All other reagents and chemicals used were of analytical grade and used without additional purification.

2.2. Preparation of Ni-P-PTFE coatings

In this study, the electrodeposition process was carried out in a 100 ml glass beaker after preparing the substrates of St37 steel and the electrodeposition solution. The anode, which is the positive pole of the electrodeposition bath, was selected from nickel with a geometric area of 20 cm², and the prepared substrates were used as the cathode, which would be the negative pole of the cell. The temperature of the plating baths was set in the range of 40-60 °C. The conditions for the electrodeposition bath used to apply the coatings are presented in Table 1. Each of the cut plates measuring one square centimeter was soldered to a 15 cm long copper wire. To avoid errors during corrosion and electrodeposition tests, the cut parts were mounted in special molds using liquid polyester. After tightening the mount and removing the electrodes from the mold, the present electrodes were mechanically sanded with

Table 1. Chemical composition and conditions of Ni-P-PTFE plating bath

Cathode	Plates st37
Time (minutes)	30
pH	8-9
Temperature (°C)	45-55
CTAB content	0.033 g/g PTFE
PTFE content	30-0 g/L
Boric acid content	45 g/L
Nickel chloride content	45 g/L
Nickel sulfate content	200 g/L
Sodium hypophosphite monohydrate	25 g/L

600, 800, 2500, and 3000 sandpapers, respectively, until their surface reached a mirror finish.

After the initial preparation, the electrodes are washed in a solvent solution that contains sodium phosphate metahydrate at a concentration of 20 g/L, sodium carbonate at a concentration of 30 g/L, soda at a concentration of 30 g/L, and sodium silicate at a concentration of 15 g/L, at a temperature of 60 °C for 15 minutes and then they were placed in 10 % weighted hydrogen peroxide solution and sulfuric acid 10 % weighted for acid washing. In the deoiling stage, the prepared electrodes were placed in the solvent solution for 15 minutes. Then, the electrodes were washed with distilled water and placed in an acid washing solution for 15 seconds to activate their surface.

2.3. Measurements

To study the morphology and substructure of the coatings, the field emission scanning electron microscope (FESEM) MIRA3 FEG-SEM, manufactured by Tescan, Czech Republic, was used at different magnifications. Through energy dispersive spectroscopy (EDS) analysis, the percentage of elements in the coatings was also determined. The micro-hardness test was conducted using the Z-HV1000 device from PACE Technologies Company, employing a Vickers indenter at the center of the coatings' cross-section under a load of 50 grams for 15 seconds. For each sample, three Vickers results were obtained, and the average of these three values was reported as microhardness. The wear test was performed using a pin-on-disc machine of Nasr Sanat Equipment Company (TSN WTC-03 model) based on the ASTM G99 standard. The applied force was 500 grams, the disk rotation speed was 95 rpm, and the travelled distance was 200 meters. The pin used is 52100 steel with 64 Rockwell C hardness. The wear test conditions were consistent across all samples, and the testing was conducted at room temperature. The friction coefficient is determined by measuring the radial force applied to the sensor and continuously recording the data with a computer.

To determine the corrosion rate of coatings, two methods of electrochemical impedance and Tafel polarization were used. In the polarization method, the working electrode is polarized by applying a potential on both sides of the anodic and cathodic potential of the open circuit. The working electrode for the electrodeposition steel sample was placed in a 100 ml beaker containing 3-5 % NaCl by weight. A platinum electrode served as the auxiliary electrode, while a calomel electrode was used as the reference electrode.

The potential curves were constructed based on the logarithm of the current using the Origa Flex device, which was equipped with the Origa Master 5 software. This was done at a scanning speed of 1 mV/s. The curves were then analyzed with the same software to determine the anodic and cathodic slopes, as well as the corrosion potential and corrosion current density. To study EIS, alternating signals with a potential range of ±5 mV were applied to the working electrode within the frequency range of 10 MHz to 100 kHz. The impedance of the sample was measured using the Origa Flex device, and the real and imaginary components of the impedance within the specified frequency range were plotted as a Nyquist diagram. In this method, a three-electrode cell was utilized for the polarization method, and the data were analyzed using Zview II software.

3. Results and discussion

Figure 1 shows the surface morphology of Ni-P coating and Ni-P-PTFE composite coatings that were deposited from solutions containing 10, 20, or 30 g/L of PTFE. It has been suggested that a five-step mechanism exists for the co-deposition of composite particles alongside metals. In the first step, the PTFE particles in the solution absorb the ionic species. In the second and third steps, particles are transported to the cathode through convection and diffusion. In the fourth step, these particles are absorbed onto the surface of the cathode while still being surrounded by a cloud of ions. Finally, in the last step, the particles are incorporated

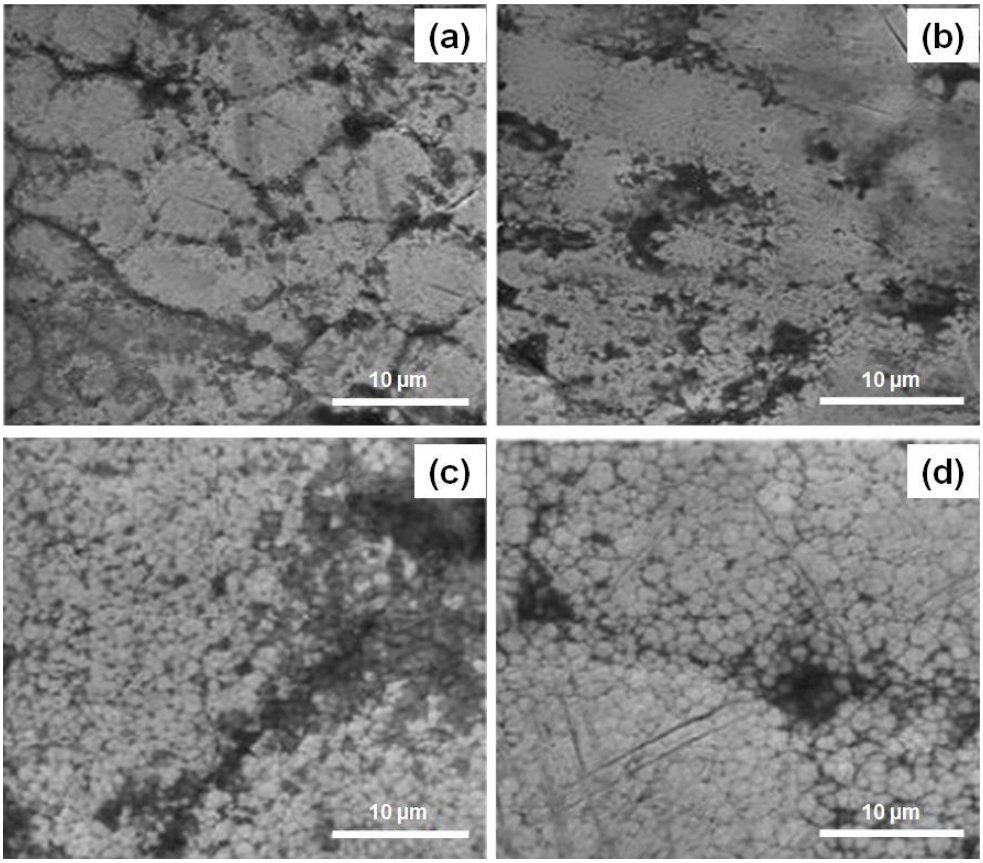

Figure 1. FESEM images of Ni-P-PTFE coatings in baths containing different concentrations of PTFE: (a) 0 g/L, (b) 10 g/L, (c) 20 g/L, and (d) 30 g/L.

into the metal network as a result of the regeneration of certain metal cations [16].

The difference between the surface morphology of Ni-P and the composite containing PTFE is that the particle size of the Ni-P coating is larger, and more intergranular boundaries are observed (Figure 1a). In the case of Ni-P-PTFE composite coatings, the coating's surface exhibits greater uniformity, smaller grain boundaries, and a reduced particle size. However, it is expected that the corrosion resistance of these coatings will increase. The images of nanocomposite coatings indicate that as the concentration of PTFE increases, and as PTFE particles fill the boundaries between the grains in the coating, the surface becomes more uniform. This uniformity is expected to reduce the penetration of corrosive solutions into the substrate. As the concentration of uniformity increases, a point is reached where excessive accumulation of PTFE particles on the surface occurs. This accumulation reduces the uniformity of the coatings, resulting in the formation of islands of Teflon polymers on the surface. Consequently, this leads to an increase in surface roughness.

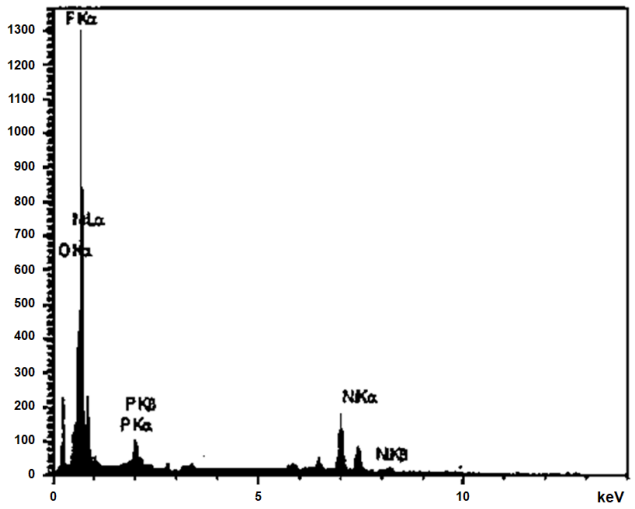

Figure 2. EDS spectrum of Ni-P-PTFE composite coating (20 g/L PTFE)

Table 2. Weight percentage of elements present in Ni-P and Ni-P-PTFE composite coatings

Samples	PTFE content	Ni (%wt)	F (%wt)	O (%wt)	P (%wt)	C (%wt)
Ni-P	-	91.54	-	0.56	9.02	-
Ni-P-PTFE-1	10 g/L	69.35	3.67	3.95	6.81	20.17
Ni-P-PTFE-2	20 g/L	61.96	4.96	1.07	6.22	26.86
Ni-P-PTFE-3	30 g/L	74.59	3.77	2.83	7.25	11.56

Among the nanocomposite coatings, the concentration at which the coating becomes uniform is found to be at PTFE 20 g/L; beyond this point, the uniformity of the coating decreases with higher concentrations (Figure 1c). EDS analysis was performed for Ni-P coatings and Ni-P-PTFE composite coatings deposited from a solution with different concentrations of PTFE. For example, the presence of peaks related to elements F and C in the spectrum related to the coating obtained from the bath containing 20 g/L of PTFE particles confirms the presence of PTFE particles in the field of Ni-P coatings (Figure 2).

Table 2 shows the percentage of elements present in the deposited coatings. It can be seen that the Ni-P coating contains about 9% by weight of phosphorus. Also, in the presence of 10 g/L of nanoparticles, the amount of phosphorus decreases by 6.22, and the measured amount of fluorine element is 3.67. Increasing the concentration of PTFE nanoparticles from 10 g/L to 20 g/L results in a higher amount of fluorine (F) in the coating. However, when the concentration is raised to 30 g/L, the nanoparticles begin to clump together in the bath. This clumping reduces the ability of the particles to integrate into the crystalline structure of the coating, leading to a decrease in the amount of fluorine detected by EDS analysis.

Ni-P coatings include phosphorus, which results in either an amorphous or nanocrystalline structure, depending on the phosphorus content and deposition conditions. Their corrosion resistance surpasses that of nickel coatings. According to the mentioned table, there is an optimal value for PTFE in Ni-P and Ni-P-PTFE coatings, which is 20 g/L, where smaller crystals are formed due to rapid germination. As a result, a smoother and shinier coating is obtained. Increasing the concentration of PTFE after the optimal point in the bath

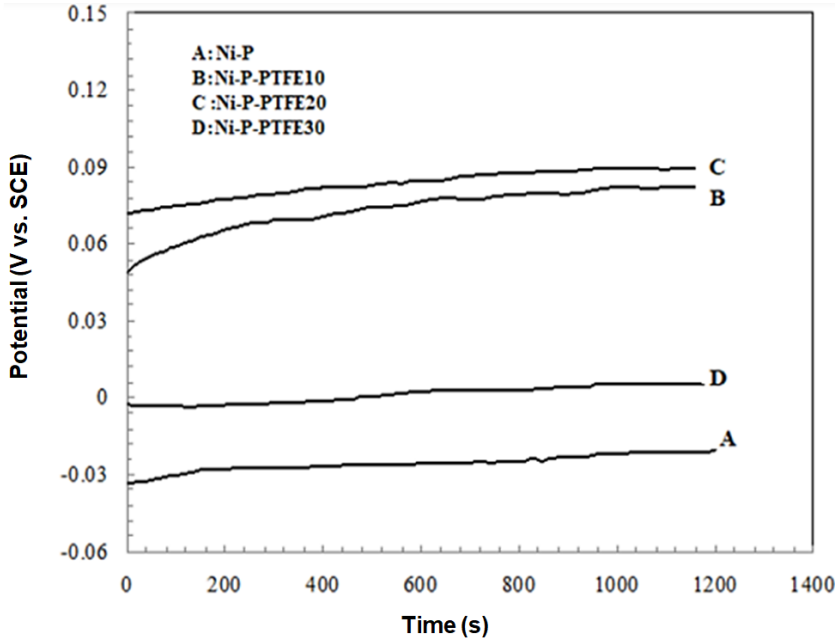

Figure 3. Diagram of open circuit potential changes with time of Ni-P coatings and Ni-P-PTFE nanocomposite in 3.5 %wt NaCl solution.

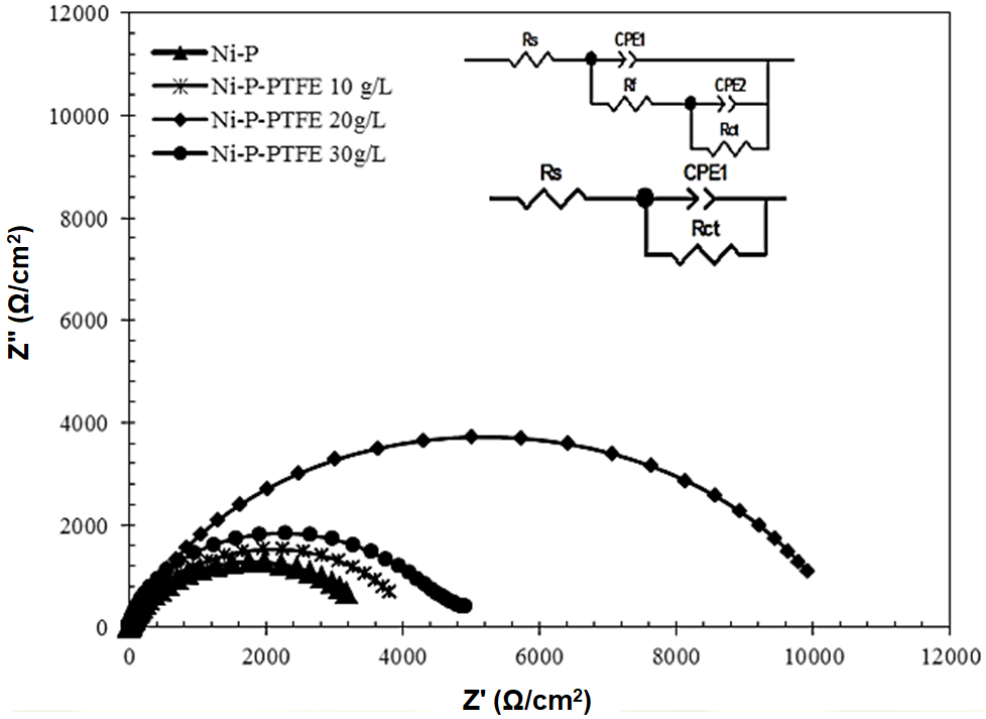

Figure 4. Nyquist diagrams for Ni-P coatings and Ni-P-PTFE composite coatings in 3.5 %wt NaCl solution.

leads to a decrease in the deposition rate. Figure 1 shows that the surface of the Ni-P coating is rougher than that of the Ni-P-PTFE coating, exhibiting extrusions of varying dimensions, with diameters ranging from a few micrometres to several nanometers. But the dimensions of the extrusions in the Ni-P-PTFE coating were smaller than the Ni-P coating.

Figure 3 shows the changes in open circuit potential (OCP) with time for Ni-P and Ni-P-PTFE composite coatings in 3.5 %wt NaCl solution. With an increase in PTFE, the open circuit potential of the samples becomes more positive, likely due to the enhanced corrosion resistance of the resulting composite coatings compared to Ni-P coatings. Various mechanisms have been proposed for the high corrosion resistance of nickel-phosphorus coatings, for example (1) the formation of a protective layer of nickel phosphate that acts as a barrier against the penetration of corrosive solutions, (2) the absorption of hypophosphite ions and the formation of a protective layer that prevents the dissolution of nickel atoms on the surface (3) with the initial dissolution of nickel atoms, a phosphorus-rich layer is created on the surface, which prevents the dissolution of nickel from the underlying layers [17].

The data indicate that the composite coating made from a bath containing 20 g/L of PTFE demonstrates the highest open circuit potential (OCP) value. This indicates that this coating has superior corrosion resistance compared to the others. As summarized in Table 2, the maximum concentration of co-deposited PTFE in the composite coatings is 20 g/L. This addition enhances the open circuit potential of the coating by forming a protective barrier, which prevents corrosive ions from penetrating the base coating and the substrate. The increase in the open circuit potential of metal coatings due to the composite with nanoparticles has also been observed in other research [18].

Figure 4 presents the Nyquist diagrams and the equivalent circuit for Ni-P coatings and Ni-P-PTFE nanocomposites in a 3.5% wt NaCl solution. The two proposed equivalent circuits exhibit the best alignment with the experimental results. By analyzing the impedance data presented in Table 3 along with the Nyquist diagrams, we can conclude that the inclusion of PTFE significantly affects the load transfer resistance (Rct), which reflects the coating's resistance to corrosion. Specifically, when the concentration of PTFE increases from 10 to 20 g/L, Rct increases sharply. However, with

Table 3. The values of the equivalent circuit elements obtained from fitting the diagrams related to the Nyquist diagram for Ni-P coatings and Ni-P-PTFE composite coatings in 3.5 %wt NaCl solution

Element	Ni-P	Ni-P-PTFE (10 g/L)	Ni-P-PTFE (20 g/L)	Ni-P-PTFE (30 g/L)
R_s	7.572	7.376	10.41	7.603
CPE_1-T	0.0000562	0.0000389	0.0000108	0.0000309
CPE_1-P	0.792	0.897	0.893	0.872
R_2	3575	267.5	324.9	251
CPE_2-T	-	0.0000739	0.0000193	0.000213
CPE_2-P	-	0.7703	0.712	0.0438
R_3 (Ω cm²)	-	3890	10141	5569

further increases in nanoparticle concentration beyond this point, the values of Rct gradually decrease.

In general, due to the smoothness of nanocomposite coatings compared to Ni-P coating, the load transfer resistance values for all nanocomposite coatings are higher than Ni-P coating. It can be suggested that composite coatings have increased corrosion resistance compared to pure Ni-P alloy coatings. The first reason is that the co-deposition of PTFE particles results in a uniform and dense coating. This density minimizes cracks and voids in the coating, preventing the electrolyte from penetrating and accelerating the corrosion of the substrate metal. The second reason is that PTFE is a non-polar particle and hence, the presence of these particles in the coating reduce the availability of the active metal surface to the corrosive solution and, as a result, the corrosion resistance of the composite increases [15].

The polarization curve of Ni-P coatings and Ni-P-PTFE composite coatings in 3.5 %wt NaCl solution is shown in Figure 5. Increasing the concentration of PTFE has decreased the corrosion current density of

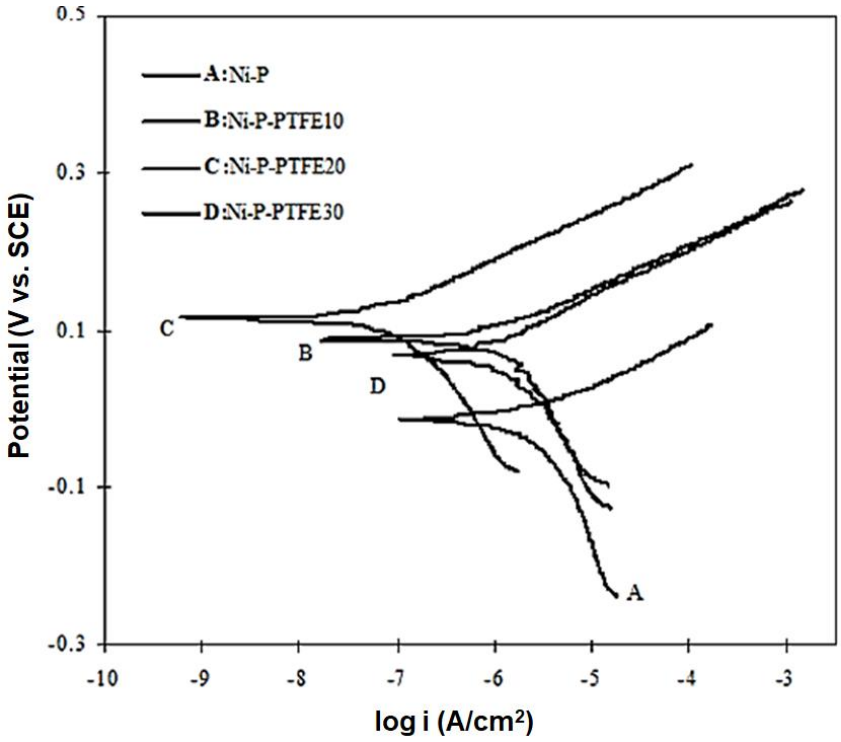

Figure 5. Tafel polarization curves of Ni-P and Ni-P-PTFE coatings in 3.5 %wt NaCl solution with a scan speed of 0.2 mV/s.

Table 4. Values of i_{corr} and E_{corr} of Ni-P and Ni-P-PTFE composite coatings in 3.5 %wt NaCl solution

Samples	E_{corr} (mV vs. SCE)	i_{corr} (A cm^{-2})
Ni-P	-11.23	5.0×10^{-6}
Ni-P-PTFE (10 g/L)	89.06	1.0×10^{-6}
Ni-P-PTFE (20 g/L)	116.47	1.0×10^{-7}
Ni-P-PTFE (30 g/L)	69.92	1.0×10^{-6}

nanocomposite coatings, shifted the corrosion potential to more positive values, and enhanced the corrosion resistance of these coatings. Table 4 shows the corrosion potential (E_{corr}) and corrosion density (i_{corr}) of these coatings. Data indicate that the lowest corrosion current and highest corrosion resistance are associated with the coating applied from a solution containing 20 g/L of polytetrafluoroethylene. In this concentration, the nucleation is fast, and the crystal growth rate is reduced. This itself makes the coating smoother and more uniform and increases its corrosion resistance compared to other coatings. The results of polarization measurements support the findings from the EIS studies.

Microhardness results shown in Figure 6 indicate that as the concentration of PTFE in the coating increases, the hardness initially decreases to a minimum value before beginning to rise again. In optimal amounts (concentration of 20 g/L) due to the presence of the largest amount of PTFE particles in the structure of the micro-hardness coating, the hardness has decreased, and in the concentration of 30 g/L of PTFE, it has increased again due to the decrease in the number of particles in the micro-hardness coating. Soft particles, such as PTFE and PVDF, reduce the hardness of composite coatings. As the volume fraction of these lubricant particles in the coating increases, the hardness decreases. This reduction in hardness contributes to an increase in the plastic deformation of the coating, primarily due to the presence of the softer particles. This effect has been documented in studies examining microhardness changes in Ni-Cu-P-PTFE coatings deposited through the electroless method [18].

To evaluate the wear resistance and friction coefficient of coatings created with varying concentrations of PTFE, a pin-on-disk test was conducted on the samples. The results of this test to investigate the effect of PTFE concentration on wear and friction coefficient are given below. Figure 7 shows that the lowest friction coefficient is associated with the composite coating derived from a 20 g/L PTFE bath. As expected, with the addition of reinforcing particles inside the composite coating, the

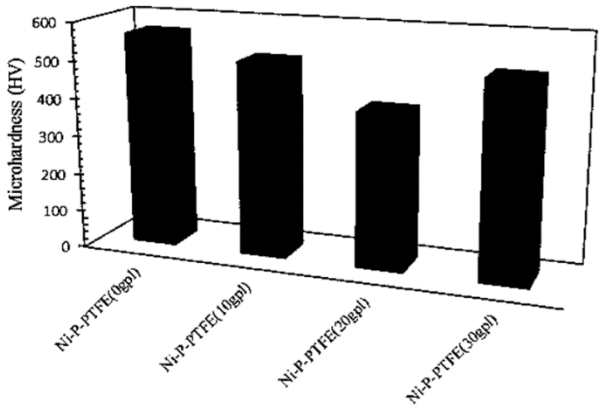

Figure 6. The effect of the concentration of PTFE particles in the plating bath on the microhardness of the Ni-P-PTFE coating.

friction coefficients have decreased from about 0.6 in the absence of PTFE particles to about 0.15 in the composite coating obtained from the 20 g/L PTFE bath. PTFE, being a soft particle, serves as a solid lubricant, effectively reducing the friction coefficient. This issue has also been observed concerning the reduction of the hardness of composite coatings, with the lowest hardness value obtained at a concentration of 20 g/L.

In the presence of 10 g/L of PTFE particles, the decreasing trend of the friction coefficient has been observed to a value of 0.4. In the presence of 30 g/L, the agglomeration of particles in the plating bath and the accumulation of PTFE particles in various areas of the coating have led to an increasing trend in the friction coefficient. In general, it can be stated that one of the factors of changes in wear resistance is the friction coefficient, and the interaction of two factors, hardness and friction coefficient, causes changes in wear resistance. Analysis of the friction coefficients indicates that the coating with 20 g/L of PTFE exhibits the best wear resistance. Similarly, improved wear resistance of electroplated Bronze coatings in the presence of PTFE particles has also been reported [19].

4. Conclusions

Ni-P-PTFE coatings were fabricated using electroplating in a watt bath, incorporating the cationic surfactant CTAB and varying concentrations of PTFE. It was observed that the morphology and electrochemical properties of these coatings depend on the amount of PTFE particles. Electrochemical and tribological studies

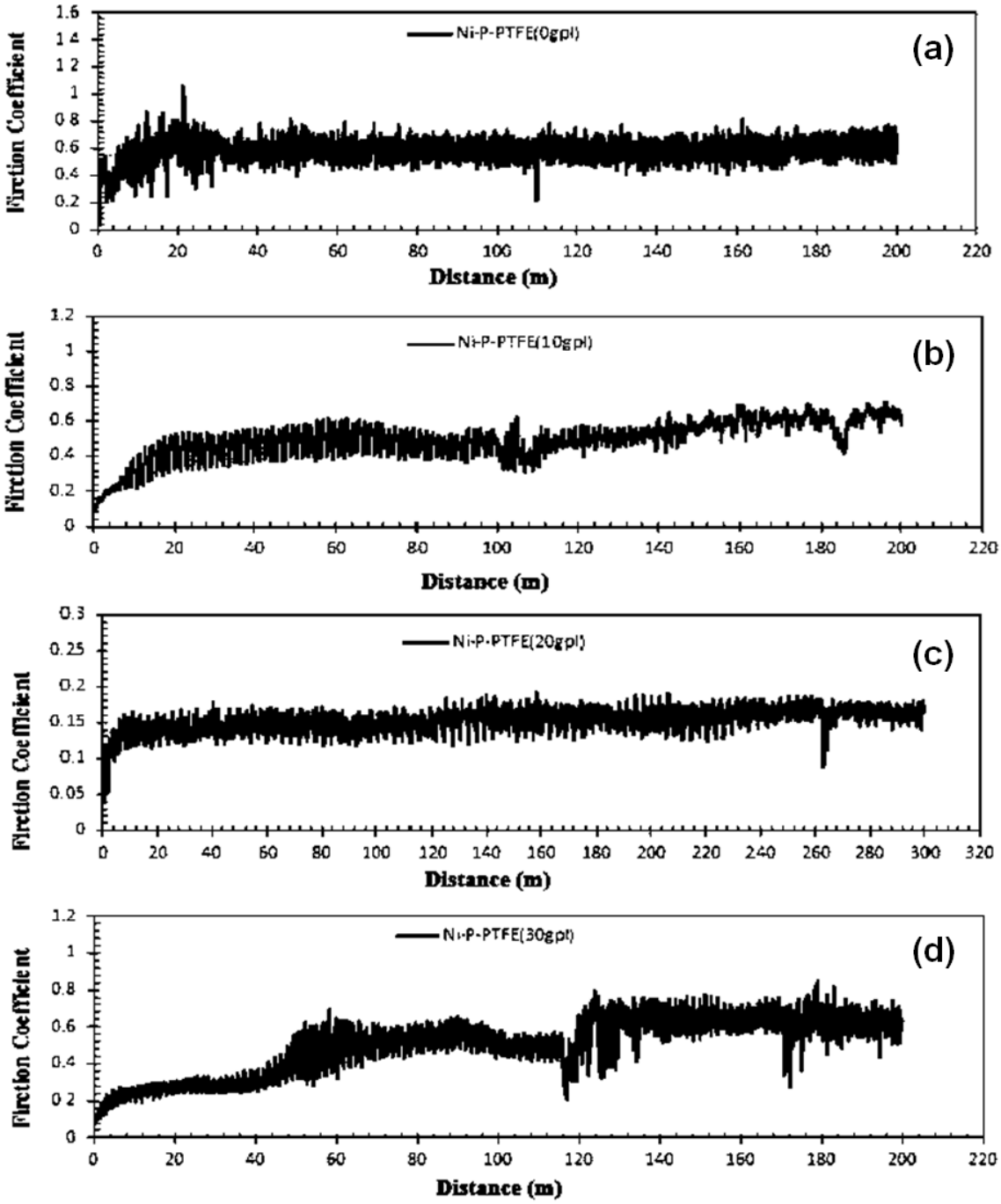

Figure 7. Changes in the friction coefficient and wear resistance of Ni-P-PTFE coating containing different amounts of PTFE particles according to wear distance: (a) 0 g/L, (b) 10 g/L, (c) 20 g/L, and (d) 30 g/L.

revealed that Ni-P-PTFE composite coatings, with a concentration of 20 g/L of PTFE particles, exhibit the highest corrosion resistance in terms of electrochemical performance. The investigation into the microhardness of the coatings indicated that the Ni-P-PTFE coating exhibited the lowest microhardness at an optimal PTFE concentration of 20 g/L. By using the pin on disc test, the lowest coefficient of friction was obtained in Ni-P-PTFE

composite coatings at a concentration of 20 g/L, and with an excessive increase of these values due to particle clumping and aggregation (agglomeration) of PTFE particles, the coefficient of friction increases.

Conflict of Interest

The authors declare no conflict of interest.

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