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Research Paper

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

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ABSTRACT

Coating is one of the effective ways to increase the corrosion resistance and wear of metallic substrates. Additionally, the composite coatings using nanoparticles can also further protect the substrate. In this study, using electrodeposition process and polytetrafluoroethylene (PTFE) particles (with concentrations of 10, 20 or 30 g /L), Ni- PTFE coatings were prepared and their corrosion and wear properties were investigated and compared with Ni-P coating. Using scanning electron microscopy (SEM) and X-ray diffraction (EDS) method, the surface morphology and elemental composition of the coatings were analyzed and finally, by using open circuit potential (OCP) techniques, electrochemical impedance spectroscopy (EIS) and TAFEL polarization techniques, the corrosion resistance of the resulting coatings in 3/5 wt.% NaCl solution were 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 SEM and EDS studies confirmed the formation of nanocomposites. Electrochemical studies also showed that Ni-PTFE coatings, at a concentration of 20 g/L PTFE, had the highest electrochemical corrosion resistance. Microhardness also decreased with increasing PTFE particles in the coating and reached its lowest value. By using the wear test, the lowest coefficient of friction obtained in composite coatings with concentration of 20 g/L, which shows the applicability of PTFE particles as a solid lubricant in Ni-P coatings.

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1. Introduction

Applying various types of protective coatings is one of the ways to reduce costs caused by corrosion and wear in different industries [1]. Among the types of coating methods such as electroless or phosphating, electrodeposition and creating different coatings such as nickel 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]. One of the most important solutions to improve properties is to add secondary elements such as iron [4] or phosphorus [5] or composite the coating by introducing micron-sized particles or Nano like SiC to the field of nickel coating [6, 7]. For example, it has been reported that by adding 10 g/L of graphene oxide to the nickel plating bath, the corrosion rate has decreased by 1/5 times [8]. The simultaneous presence of Iron element and TiC ceramic particles in the structure of the nickel coating also causes a corrosion current density of 0/79 microamps per decimeter [9].

Various studies have shown that wear and friction are of complex processes that their values depend on the surface properties of the two slippery objects, tribological conditions and their surrounding environment. Many dynamic systems are lubricated by different lubricating fluids. But one of the disadvantages of lubricating liquids is the inability to use them at high temperatures or under vacuum, because such materials generally lose their performance in these conditions. Therefore, most of the research has been focused on another category of materials that reduce the friction coefficient and are called solid lubricants [10, 11].

In recent years, many researches have been conducted on the possibility of using particles such as WS_2 and MoS_2 as solid lubricants in coatings [12, 13]. It has been observed that if solid lubricants are used on the surface of parts under slippery, a layer between two surfaces in slippage is created and prevents the contact and welding of their surface heterogeneities with each other. Some solid materials have low shear strength and when placed on a slippery surface, they greatly reduce wear and friction, because the use of solid lubricants in the form of protective coatings seems necessary [14, 15]. For example, research results have demonstrated that in the presence of WS_2, the friction coefficient of Ni-P coatings obtained by 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, which its effect on reducing the friction coefficient of various coatings such as Ni-W has been studied [16]. However, the effect of the presence of this material in the field of Ni-P coatings has not been comprehensively studied. Also, the optimal concentration of PTFE under which the maximum amount of corrosion resistance or wear resistance and hardness can be observed has not been investigated yet.

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. Next, the effect of participation percentage of PTFE in the coating on the corrosion behavior of the coatings was studied using TOEFL polarization test and electrochemical impedance spectroscopy (EIS) in the environment of 3/5% 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.

2. Experimental Section

2.1. Materials Synthesis

In this study, the electrodeposition process was carried out in a 100 ml glass beaker after preparing the substrates of st37 steel and electrodeposition solution. The anode, which is the positive pole of the electrodeposition bath, was selected from nickel with a geometric area of 20 square centimeters, 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 of the electrodeposition bath to apply the coatings are shown in Table 1. Each of the cut plates measuring one square centimeter was soldered to a 15 cm long copper wire. In order to avoid test errors during corrosion and electrodeposition tests, the cut parts were also mounted by liquid polyester in special molds. After tightening the mount and removing the electrodes from the mold, the present electrodes were mechanically sanded with 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 20g/L, sodium carbonate at a concentration of 30 g/L, soda (30 g/L), and sodium silicate (15 g/L) at 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 by distilled water and placed in acid washing solution for 15 seconds to activate their surface.

2.2. Test Methods

Different methods were used to check the properties of the coatings. To study the morphology and substructure of the coatings, the field emission scanning electron microscope (FESEM) model MIRA3 FEG-SEM, manufactured by Tescan, Czech Republic, was used in different magnifications. Through EDS analysis, the percentage of elements in the coats were also determined. The micro-hardness test was performed using the Z-HV1000 device of PACE Technologies Company with a Vickers indenter in the middle of the cross section of the coatings under an applied load of 50 grams for 15 seconds.

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	0/033g/g PTFE
PTFE	30-0 g/L
Boric acid	45 g/L
Nickel chloride	45 g/L
Nickel sulfate	200 g/L
Sodium hypophosphite monohydrate	25 g/L

For each sample, 3 Vickers results were created and the average of these 3 numbers 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 ASTMG99 standard. The applied force was 500 grams, the disk rotation speed was 95 rpm, and the traveled distance was 200 meters. The pin used is 52100 steel with 64 Rockwell C hardness. The wear test conditions were the same for all samples and the wear was done at ambient temperature. The friction coefficient is measured by measuring the radial force applied to the force sensor and its continuous recording by the computer. To determine the corrosion rate of coatings, two methods of electrochemical impedance and TOFEL 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 of the electrodeposition steel sample was considered to be placed inside a 100 ml beaker containing 3/5% weight of NaCl Platinum electrode and calomel electrode were used as auxiliary and reference electrodes, respectively. The potential curves were drawn according to the logarithm of the current using the Origa Flex device equipped with the Origa Master5 software with a scanning speed of 1 mV/s, and then the curves were analyzed using the same software to find the anodic and cathodic slopes, the corrosion potential and corrosion current density. In order to study EIS, alternating signals with a potential range of $\pm$5mV were applied to the working electrode in the frequency range of 10 MHz to 100 kHz. The impedance of the sample was drawn by the Origa Flex device and the real and imaginary components of the impedance in the mentioned frequency range in the form of a Nyquist diagram. In this method, the three-electrode cell used in the polarization method, and Zview II software was used to analyze the data.

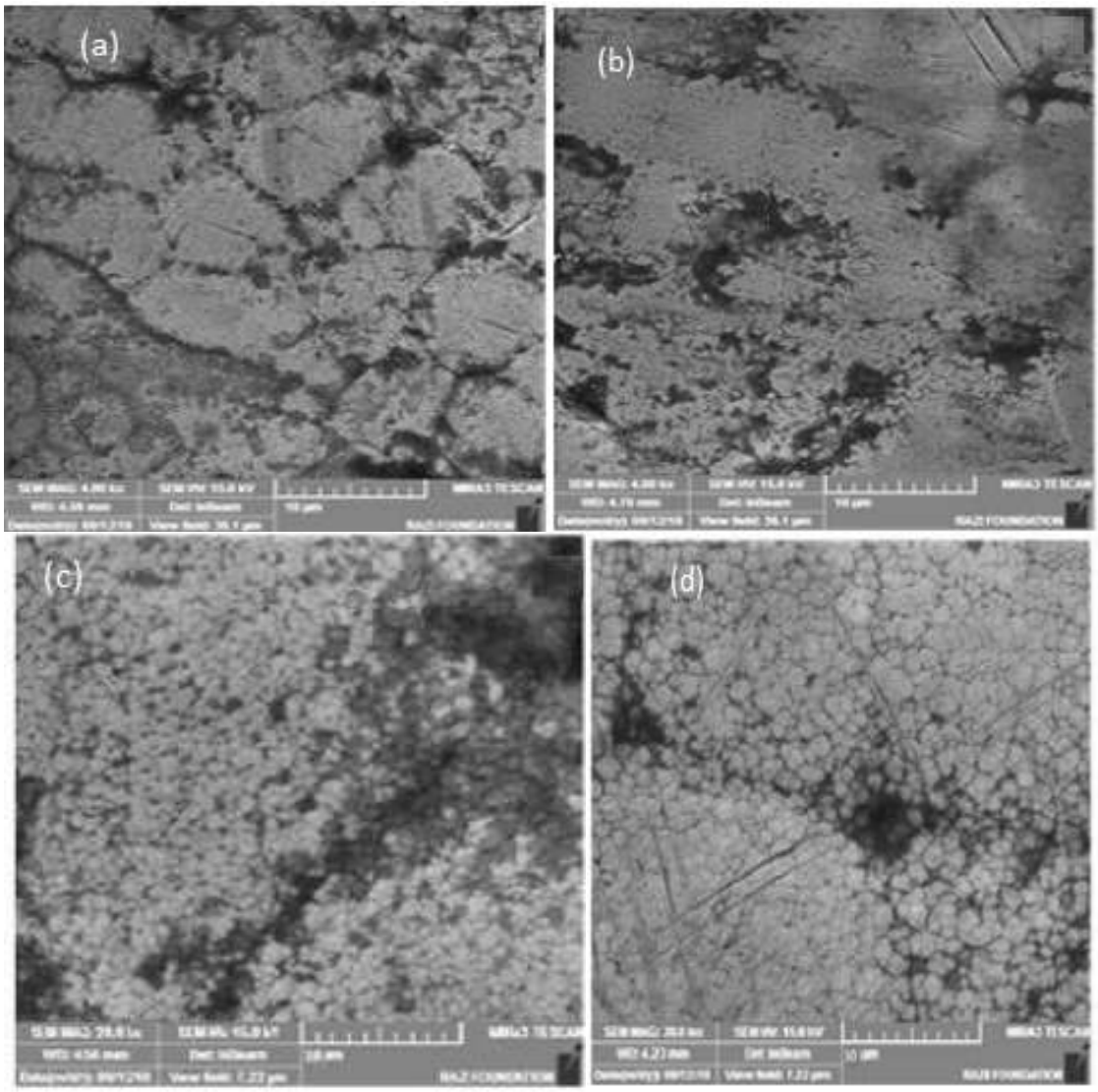


Fig. 1. SEM images of Ni-P-PTFE coatings in baths containing different concentrations of PTFE: a) 0 g/L. b) 10 g/L. c) 20 g/L. d) 30 g/L.

3. Results and Discussion

3.1. Investigation of Surface Morphology and Elemental Analysis of Ni-P-PTFE Coatings

Figure 1 illustrates 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 proposed that there is a five-step mechanism for co-deposition of composite particles with metals. In the first step, the PTFE particles in the solution absorb the ionic species. In the second and third steps, these particles are transported to the cathode by convection and diffusion. In the fourth step, they are absorbed on the surface of the cathode, so that they are still surrounded by ion cloud, and in the last step, the particles are absorbed as a result of the regeneration of some metal cations and are placed in the metal network [17]. The difference between the surface morphology of Ni-P and the composite containing of PTFE is in that the particle size of the Ni-P coating is larger and more inter-granular boundaries are observed (Figure 1a). In the case of Ni-P-PTFE composite coatings, it can be seen that the surface of the coating is more uniform, the grain boundaries are smaller, and the particle size is smaller. However, it is expected that the corrosion resistance of these coatings will increase. It can be seen from the images of nanocomposite coatings that with the increase in the concentration of PTFE and then the filling of the boundaries between the grains in the coating with PTFE particles, the surface becomes uniform and it is expected that the penetration of the corrosive solution into the substrate will decrease. Moreover, certain concentration of this uniformity increases up and after that, due to the excessive accumulation of PTFE particles on the surface, the uniformity of the coatings is reduced and the surface of the coating is covered with islands of Teflon polymers, which causes surface roughness. According to the pictures, among the nanocomposite coatings, the concentration to which the coating becomes uniform

and after that, the uniformity of the coating decreases with concentrations of PTFE 20 g/L (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. By increasing the concentration of PTFE nanoparticles from 10 g/L to 20 g/L, the amount of F element increases in the coating, but increase of concentration of PTFE nanoparticles in the bath to 30 g/L, causes clumping of particles in the bath, which has reduced the entry of particles into the crystalline structure of the coating and as a result, the amount of element F detected by EDS analysis has decreased. In general, due to having phosphorus in their structure, Ni-P coatings have an amorphous or nanocrystalline structure depending on the amount of phosphorus and deposition conditions, and their corrosion resistance is higher compared to Ni coating. 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, in which 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 leads to a decrease in the deposition rate. According to Figure 1, the surface of the Ni-P coating is rougher than the Ni-P-PTFE coating and has extrusion of different dimensions, which its diameter varies from a few micrometers to a few nanometers. But the dimensions of the extrusions in the Ni-P-PTFE coating were smaller than the Ni-P coating.

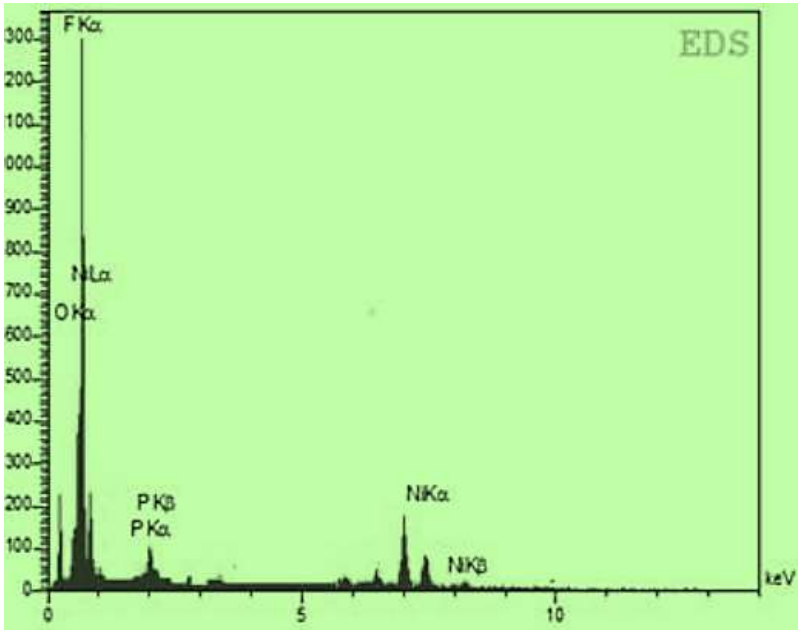


Fig. 2. EDS spectrum of Ni-P-PTFE composite coating applied from bath with 20 g/L PTFE

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

		Ni (%wt)	F (%wt)	O (%wt)	P (%wt)	C (%wt)
Ni-P		91/54	-	0/56	9/02	-
Ni-P-PTFE	10 g/L	69/35	3/67	3/95	6/81	20/17
Ni-P-PTFE	20 g/L	61/96	4/96	1/07	6/22	26/86
Ni-P-PTFE	30 g/L	74/59	3/77	2/83	7/25	11/56

3.2. Investigation of Corrosion Resistance of Ni-P-PTFE Coatings

Figure 3 shows the changes of open circuit potential (OCP) with time for Ni-P and Ni-P-PTFE composite coatings in 3/5 %wt NaCl solution. It can be seen that with the increase of PTFE, the open circuit potential of the samples becomes more positive, likely due to the enhanced corrosion resistance of the 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 [18]. Also based on this curve, the composite coating obtained from the bath containing 20 g/L of PTFE has the most positive OCP value, and this shows the high corrosion resistance of this coating compared to other coatings. According to Table 2, the maximum amount of co-deposited PTFE in the composite coatings related to the bath includes 20 g/L of PTFE, which increases the open circuit potential of the coating due to the creation of a protective barrier against the penetration of corrosive ions into the base coating and the substrate. The increase in the open circuit potential of metal coatings due to composite with nanoparticles has also been observed in other researches [19].

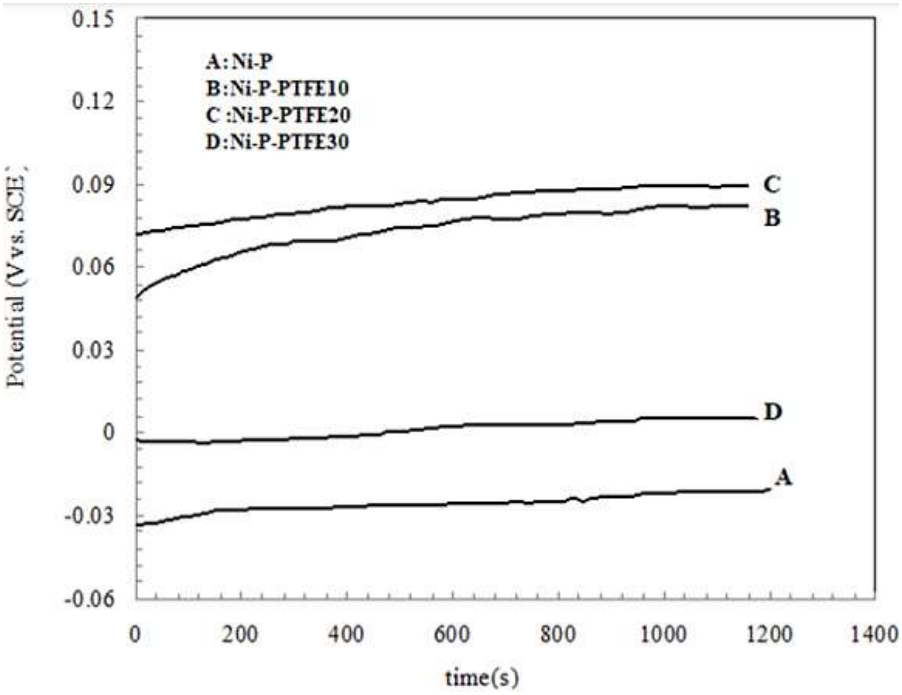


Fig. 3. Diagram of open circuit potential changes with time of Ni-P coatings and Ni-P-PTFE nanocomposite in 3/5 %wt NaCl solution

Figure 4 shows the Nyquist diagrams along with the equivalent circuit of Ni-P coatings and Ni-P-PTFE nanocomposite in 3/5 %wt NaCl solution. These two proposed equivalent circuits have the most adjustment with the experimental results. By comparing the impedance data summarized in Table 3 and the Nyquist diagrams, it can be concluded that in the presence of PTFE and by adding their concentration from 10 to 20 g/L, the load transfer resistance (Rct), which indicates the resistance of the coating against corrosion, increases sharply, and with the further increase in the concentration of nanoparticles, the values of this parameter 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 to increase the corrosion resistance of composite coatings compared to pure Ni-P alloy coatings. The first reason is that co-deposition of PTFE particles creates a uniform and dense coating, so that the cracks and voids of the coating are very small and the electrolyte cannot pass through the coating and accelerate the corrosion of the substrate metal. The second reason is that PTFE is a non-polar particle, so by the presence of these particles in the coating, less active metal surface is available to the corrosive solution, and as a result, the corrosion resistance of the composite increases [16].

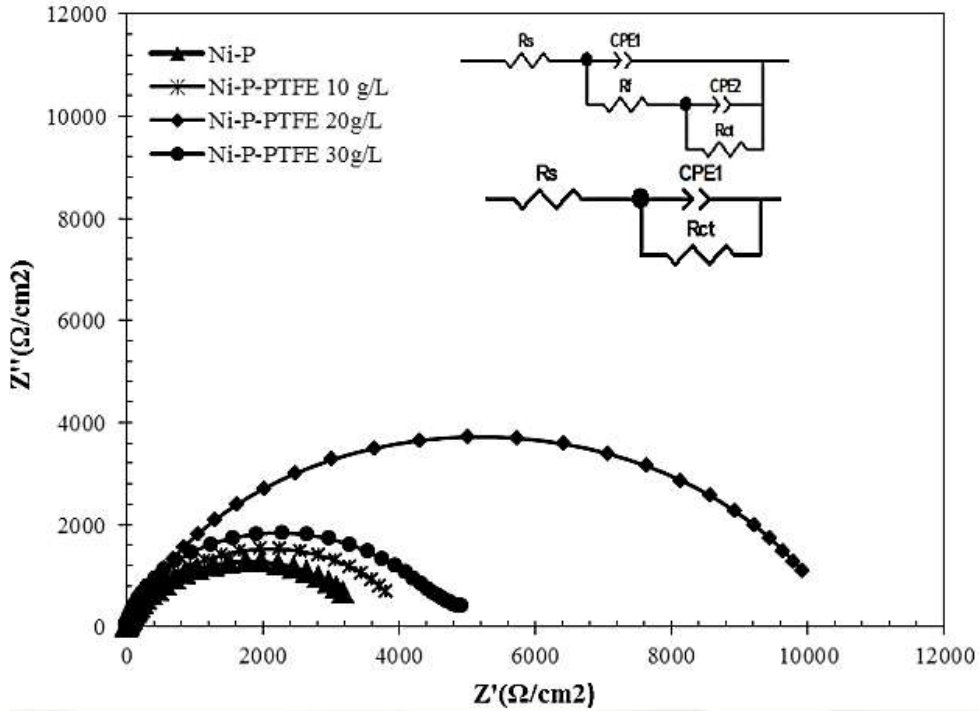


Fig. 4. Nyquist diagrams for Ni-P coatings and Ni-P-PTFE composite coatings in 3/5 %wt NaCl solution

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 cm^2)	-	3890	10141	5569

3.3. TOFEL Polarization Curves

The polarization curve of Ni-P coatings and Ni-P-PTFE composite coatings in 3/5 %wt NaCl solution is shown in Figure 5. As seen in the figure, increasing the concentration of PTFE has reduced the corrosion current density of nanocomposite coatings and shifted the corrosion potential to more positive values and increased the corrosion resistance of these coatings. Table 4 shows the corrosion potential (E_{corr}) and corrosion density (i_{corr}) of these coatings. According to the data, it can be concluded that the lowest corrosion current and the highest corrosion resistance are related to the coating deposited from the 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. In this way, the results of polarization measurements confirm the results of EIS studies.

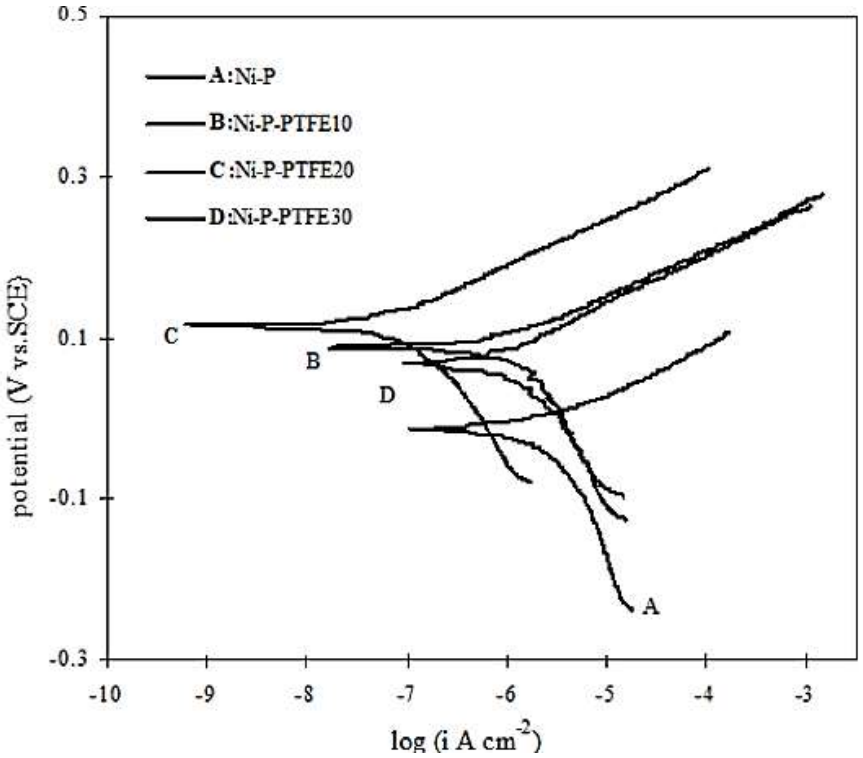


Fig. 5. TOFEL polarization curves of Ni-P and Ni-P-PTFE coatings in 3/5 %wt NaCl solution with scan speed of $0/2mVS^{-1}$

Table 4. Values of icorr and Ecorr of Ni-P and Ni-P-PTFE composite coatings in 3/5 %wt NaCl solution

	E_{corr} (mV vs. SCE)	i_{corr} (A cm^{-2})
Ni-P	-11/23	$5/0\times10^{-6}$
Ni-P-PTFE (10 g/L)	89/06	$1/0\times10^{-6}$
Ni-P-PTFE (20 g/L)	116/47	$1/0\times10^{-7}$
Ni-P-PTFE (30 g/L)	69/92	$1/0\times10^{-6}$

In according to Figure 6, the microhardness with the increase in the concentration of PTFE in the coating, the hardness first decreased and reached a minimum value, and then began an upward trend. 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 amount of particles in the micro-hardness coating. Soft particles such as PTFE, PVDF and so on, reduce the hardness of the composite coating, and the hardness decreases with the increase in the volume fraction of the lubricant particles in the coating, which is the reason for the increase in the plastic change of the coating due to the presence of soft particles in the coating. This process has been reported on microhardness changes in the presence of PTFE particles in Ni-Cu-P-PTFE coating deposited by electroless method [19].

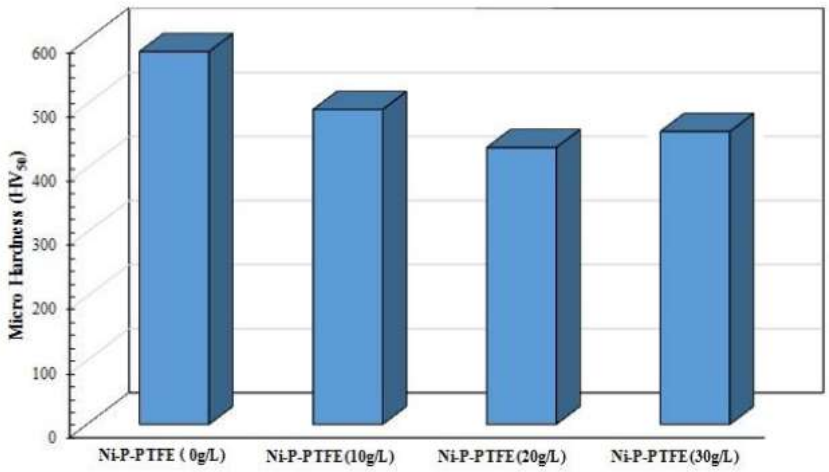
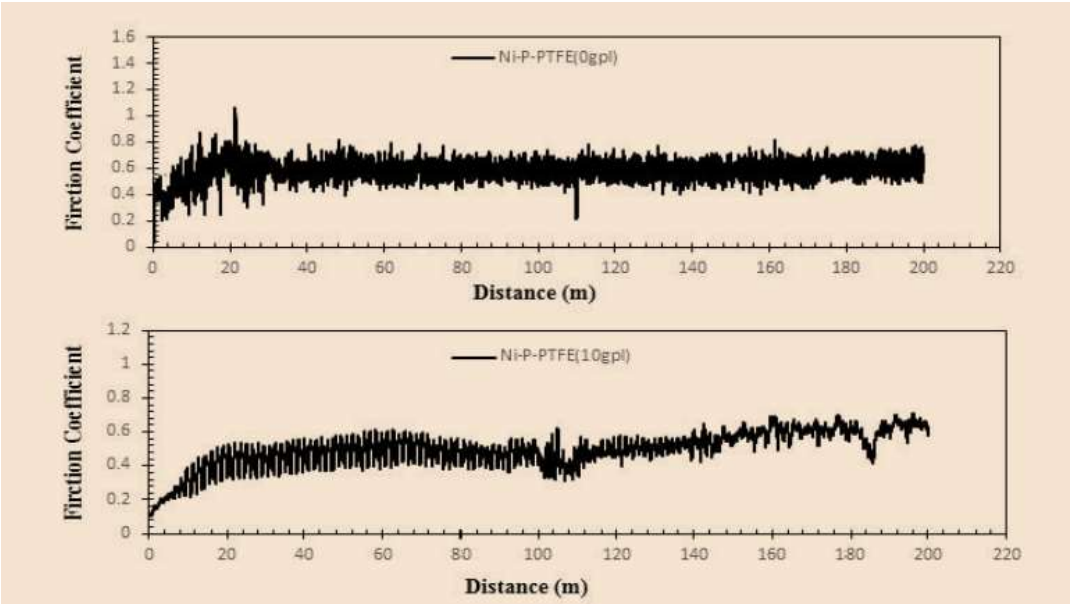


Fig. 6. The effect of the concentration of PTFE particles in the plating bath on the microhardness of the Ni-P-PTFE coating

3.4. Investigating the Mechanical and Tribological Behavior of Ni-P-PTFE Coatings

To check the wear resistance and friction coefficient of the coatings created with different concentrations of PTFE, a pin on disk test was performed on the samples. The results of this test to investigate the effect of PTFE concentration on wear and friction coefficient are given below. According to Figure 7, the lowest friction coefficient obtained is related to the composite coating of 20 g/L PTFE bath. As expected, with the addition of reinforcing particles inside the composite coating, the 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. In this regard, it can be stated that since PTFE is a soft particle, it acts as a solid lubricant and reduces the friction coefficient. This issue has also been observed regarding the reduction of the hardness of composite coatings, so that the lowest hardness value has been 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 the value of 0/4. Also, in the presence of 30 g/L, due to the agglomeration of particles in the plating bath and the accumulation of PTFE particles in different spots of coating, the friction coefficient has shown an increasing trend. In general, it can be stated that one of the factors of changes in wear resistance is friction coefficient, and the interaction of two factors, hardness and friction coefficient, causes changes in wear resistance. By putting together the results of the friction coefficient of the samples, it can be seen that the coating obtained in the presence of 20 g/L of PTFE has the best wear resistance conditions. Similarly, improved wear resistance of electroplated Bronz coatings in the presence of PTFE particles has also been reported [20].



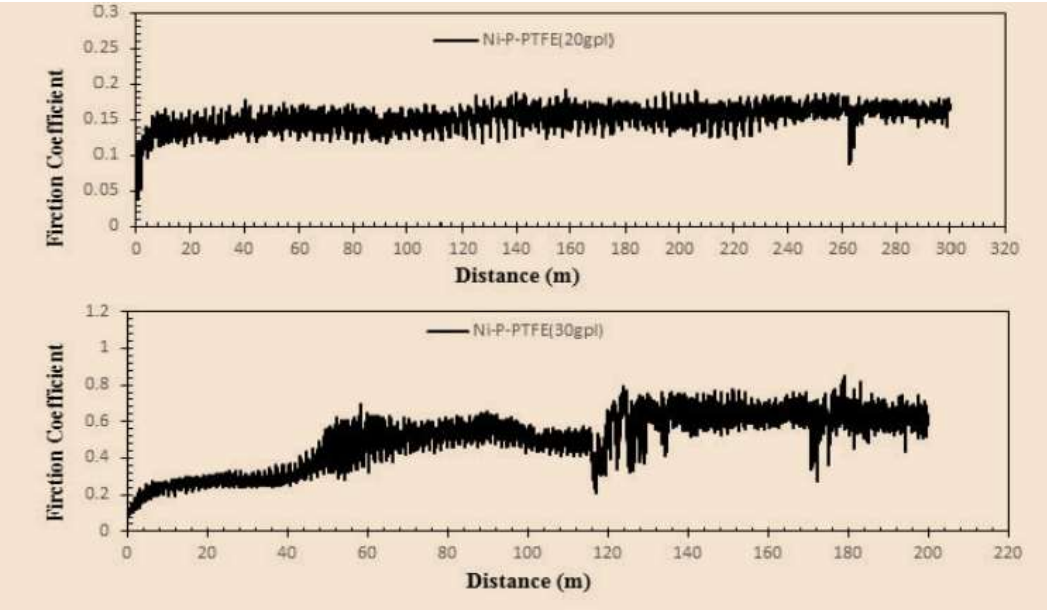


Fig. 7. Changes in friction coefficient and wear resistance of Ni-P-PTFE coating containing different amounts of PTFE particles according to wear distance.

4. Conclusion

In this study, using electroplating process and PTFE particles (with concentrations of 10, 20 or 30 g/L), Ni-P-PTFE coatings were prepared and their corrosion and abrasion properties were investigated and compared with Ni-P coating. Ni-P-PTFE coatings were prepared through electroplating in a watt bath, in the presence of CTAB active cationic surfactant factor and different 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 also showed that Ni-P-PTFE composite coatings, in concentrations of 20 g/L of PTFE particles, have the highest corrosion resistance in terms of electrochemical. The investigation of the microhardness of the coatings showed that the lowest microhardness of the Ni-P-PTFE coating was obtained at the optimal concentration of 20 g/L of PTFE. By using the pin on disc test, the lowest coefficient of friction 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 friction is increasing.

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