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Investigation of the effect of current density and ZrO_2 bath concentration on electrodeposited Ni-P/ZrO_2 composite coatings

Akım yoğunluğu ve ZrO_2 banyo konsantrasyonunun elektrodepolanmış Ni-P/ZrO_2 kompozit kaplamalar üzerindeki etkisinin incelenmesi

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Abstract

In this study, Ni-P/ZrO_2 nanocomposite coatings were deposited on St-37 steel substrates using the electroplating method with varying ZrO_2 concentrations (10 g/L ve 20 g/L) and current densities (50 mA/cm^2 ve 75 mA/cm^2). To investigate the effects of electrolyte components and current density on coating properties, analyses were performed regarding microhardness, wear performance, and surface morphology. As a result of the analyses, it is observed that different bath concentrations and current densities significantly affect properties such as morphology, hardness, and wear performance. It is seen that the surface morphologies of the obtained coatings are generally smooth, but it is understood from the optical images that the surfaces of all nanocomposite coatings are rougher. While adding ZrO_2 nanoparticles to the main matrix increases microhardness by approximately 40% compared to pure nickel, a similar but higher hardness value was obtained with the increase in current density. When examined in terms of wear performance, an average friction coefficient value of 3.15 times higher than that of pure nickel nanocomposite coatings was obtained.

Keywords: Electrodeposition, Ni-P alloy, Nanocomposite coating, Microhardness, Wear

Öz

Bu çalışmada, Ni-P/ZrO_2 nanokompozit kaplamalar, değişen ZrO_2 konsantrasyonları (10 g/L ve 20 g/L) ve akım yoğunlukları (50 mA/cm^2 ve 75 mA/cm^2) ile elektrokaplama yöntemi kullanılarak St-37 çelik alt tabakalar üzerine biriktirildi. Elektrolit bileşenlerinin ve akım yoğunluğunun kaplama özelliklerine etkilerini incelemek amacıyla mikrosertlik, aşınma performansı ve yüzey morfolojileri açısından analizler yapılmıştır. Yapılan analizler sonucunda farklı banyo konsantrasyonun ve akım yoğunluğunun morfoloji, sertlik ve aşınma performansı gibi özellikleri ciddi miktarda etkilediği görülmektedir. Elde edilen kaplamaların genel olarak yüzey morfolojiler pürüzsüz olduğu görülmektedir ama nanokompozit kaplamaların hepsinde yüzeyinin daha pürüzlü olduğu optik resimlerden anlaşılmaktadır. Ana matrise eklenen ZrO_2 nanopartikülü ilavesi mikro sertliği saf nikele göre yaklaşık %40 oranında artırırken, akım yoğunluğunun artmasıyla beraber yakın ama daha yüksek sertlik değeri elde edilmiştir. Aşınma performansı bakımından incelendiğinde, saf nikel nanokompozit kaplamalara göre 3.15 kat daha fazla ortalama sürtünme katsayısı değeri elde edilmiştir.

Anahtar kelimeler: Elektrodepolama, Ni-P alaşım, Nanokompozit kaplama, Mikrosertlik, Aşınma

1 Introduction

Low-carbon steel exhibits excellent mechanical properties as well as ease of production and recycling [1], [2]. However, its industrial applications are hampered by limitations in wear and corrosion resistance [3]. To overcome this challenge, various material surface modification techniques have been used, including electroplating, thermal spraying, vapor deposition, and non-electrolytic deposition [4]. Among these methods, electrolytic deposition stands out due to its simplicity and ability to effectively coat complex workpieces [5]. The process enhances wear resistance and corrosion protection, making it a widely adopted solution in the field of metal protection. In recent years, nickel-based coatings have been widely used in industry [6]. The Ni-P coating remains a widely used research topic among chemically deposited metals due to its extensive applicability in protective coating applications, which were first developed in the 1950s. However, the formation of defects such as pinholes and microcracks during the deposition process may result in corrosion failure of the coating, which limits its further use. Consequently, there is a significant research focus on reducing coating defects [7, 8]. Ni-P alloy coating technology has recently been important in developing these research areas. Among these research areas, promising methods, such as adding nanoparticles to the main matrix of metal coatings, can enhance protection and mechanical properties [9, 10]. Currently, nanoparticles such as Y_2O_3 (Yttrium oxide) [11], WC (Tungsten carbide) [12], Al_2O_3

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(Aluminum oxide) [13], CeO_2 (Cerium oxide) [14], SiO_2 (Silicon dioxide) [15], and others are used as reinforcement particles for metal coatings. By using different types of nanoparticles, the performance of nanocomposite coating can be positively enhanced and adjusted to be integrated into various applications [16]. A.B. Radwan et al. [17] obtained nanocomposite coatings using Ni-P/Y_2O_3 electroplating. The findings indicated that the deposition of Y_2O_3 particles on the Ni–P metal matrix exhibited superior mechanical properties. Furthermore, it was shown that the corrosion resistance of Ni–P/Y_2O_3 composite coatings was increased by up to 90.8% at 1.00 g/l Y_2O_3 bath concentrations compared to Ni–P coatings. Liu et al. [18] found that the wear resistance of Ni−P/WC composite coating was enhanced by the deposition of WC nanoparticles on a metal matrix. Furthermore, the corrosion resistance properties of the coating were also advanced by dispersing the nanoparticles in the matrix. Alirezaei et al. [19] Ni–P/Al_2O_3 composite coatings were obtained with different deposition rates. It was shown that the percentage of deposition particles, roughness, and hardness of the coating were affected by the Al_2O_3 concentration used in the bath. As a result, the average roughness and hardness values increased with adding Al_2O_3 nanoparticles. Xiao et al. [20] produced a Ni-P metal matrix alloy with a hardness of 580 HV by adding 15 g/L CeO_2 nanoparticles to the bath concentration. In recent years, zirconium dioxide (ZrO_2) as a reinforcing particle has gained wide application in industry due to its outstanding mechanical properties and chemical resistance. Its high melting point (about 2700°C) and hardness make zirconium ideal for cutting tools, abrasive materials, and durable ceramics [21]. Lehman et al. [22] synthesized Ni-W/ZrO_2 composite coatings using the electrochemical deposition method in an electroplating solution. Chemical composition, microstructure, mechanical properties, wear characteristics, and corrosion resistance of Ni-W/ZrO_2 composite coatings were optimized by changing bath parameters. In addition, the relationship between ZrO_2 powder content and current density was investigated. According to the results, the most optimum results were obtained at approximately 5 g/L zirconium nanoparticle content in the coating solution, 11 A/dm^2 current density, and 600 rpm cathode rotation speed. Accordingly, microhardness was 8 GPa, wear resistance was 1.4 x $10^{-6}$$mm^3$/Nm, and corrosion resistance was 6 µA/cm^2. Wang et al. [23] prepared two distinct Ni-ZrO_2 nanocomposite coatings via pulsed electrodeposition (PC) and pulsed reverse electrodeposition (PRC), respectively. The findings revealed that Ni-ZrO_2 nanocomposite coatings demonstrated enhanced microhardness and superior wear resistance compared to pure nickel coatings under identical conditions. Furthermore, the wear weight losses of the PC and PRC nanocomposite coatings were found to be lower than those of the DC nanocomposite coatings. It also showed that Ni-ZrO_2 nanocomposite coatings obtained by the PRC method have superior wear resistance properties. Therefore, using ZrO_2 to enhance the mechanical and wear performance of composite coatings is among the good options [24]. In addition, there are few studies on chemically deposited Ni-P/ZrO_2 nanocomposite coatings [25-27]. Therefore, the aim of this study, in addition to the studies conducted in other studies, is not only to investigate the effect of ZrO2 on the structure, corrosion resistance and microhardness of Ni-P alloy coatings, but also to expand the effect of different bath concentrations and different current densities on the host matrix. In this study, Ni-P/ZrO_2 composite coating was deposited on the surface of St-37 steel by electroplating method. The effects of different ZrO_2 bath concentrations and current density on the morphology, microhardness, and wear performance of Ni-P coatings were investigated. The mechanisms of wear of the composite coating and the causes of adverse effects were subjected to analysis.

2 Material and methods

Composite coatings with Ni-P main matrix and ZrO_2 nanoparticle reinforcement were deposited on St-37 steel substrate material by electrodeposition method with a conventional watt-type nickel bath. Sodium hypophosphite was added to the bath as a phosphate source to obtain the main matrix structure. During the preparation of the samples, a 3-electrode system was used in the electrodeposition bath. Cathode, anode, and reference electrodes were placed vertically in the bath, and approximately 3 cm was determined between them. The schematic representation of the electrodeposition system is provided in Figure 1. Ultrasonic stirring was performed for 1h at room temperature to enhance the dispersion of ZrO_2 particles used in the electrodeposition solution and to prevent their possible aggregation. Vicra Cell VCX 750 brand and model was used as the ultrasonic mixer; the cycle value was set to 1, and the amplitude was set to 70% (~20 kHz).

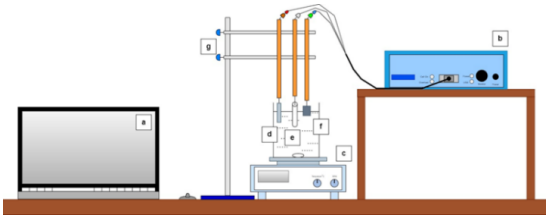

Figure 1. Electrodeposition system (a) computer (software control), (b) potentiostat/galvanostat, (c) magnetic stirrer, (d) nickel plate (anode), (e) reference electrode (Ag/AgCI), (f) St-37 (cathode), (g) stand

Ag/AgCI was used as the reference electrode, nickel plate as the anode, and St-37 steel as a cathode. Preparation of the substrate samples is one of the important processes. As the first process, the sample was sanded with 600-1200-2000# sandpapers to clean the oil, dirt, and rust layers on it. Then, the remaining area was painted with isolated paint so that 3 cm^2 of the substrate sample was exposed. After this process, a pure aqueous solution of 20% acetone was prepared and cleaned in an ultrasonic bath for 30 minutes and then kept in a pure aqueous solution of 10% hydrochloric acid (HCl) for 1 minute for etching. After each process, the samples were rinsed with pure water and left to dry. All experiments, including electrochemical corrosion tests, were performed with a Gamry interface 1010 E brand and model device. In the electrodeposition bath, nickel sulfate

hexahydrate ($NiSO_4.6H_2O$) and nickel chloride hexahydrate ($NiCI_2.6H_2O$) were used as pure nickel sources, and sodium hypophosphite was used as a phosphate source. The reinforcement particle used in the composite coating bath was 40-50 nm zirconium oxide (ZrO_2) particles. The added reinforcement particles cause agglomeration and surface elevation problems in the bath. Therefore, sodium dodecyl sulfate was added to the electroplating bath and stirred with an ultrasonic mixer for 1 h before coating; the cycle value was set to 1, and the amplitude value was set to 75%. In all coating processes, 50 and 75 mA/cm^2 current density and 2 ± 0.1 pH value was used. The bath temperature was set around 65 ± 1 ºC. The storage period was studied as 60 min. The working parameters and working conditions of all these experiments are given in Table 1. Code names have been assigned to the bath concentrations of nanocomposite coatings in Table 2. After the electrodeposition process was completed, it was cleaned with pure water and left to dry at room temperature.

Table 1. Bath parameters and conditions

Sq. no	Bath components (Chemicals)	Quantity
1	Nickel-Sulfate-Hexahydrate	240 g/L
2	Nickel-Chloride-Hexahydrate	15 g/L
3	Sodium Hypophosphite	20 g/L
4	Boric Acid	30 g/L
5	Sodium Chloride	15 g/L
6	Phosphoric Acid	6 g/L
7	Zirconium Oxide	0-10-20 g/L
8	Sodium dodecyl sulfate (SDS)	0.5 g/L
9	Saccharin	2 g/L
Sq. no	Bath conditions	Parameters
1	Electrolyte Bath pH	2 ± 0.1
2	Temperature	65 ± 0.2 °C
3	Shaking the Bath Using a Magnetic Stirrer	300 ± 50 rpm
4	Time (Coating deposition)	60 minutes
5	DC Current Density	50-75 mA/cm^2

Table 2. Code names

Parameters		Code Names
Current Density (mA/cm^2)	ZrO_2 (g/L)	
50	0	50NiP0Z
50	10	50NiP10Z
50	20	50NiP20Z
75	0	75NiP0Z
75	10	75NiP10Z
75	20	75NiP20Z

For the surface analysis, images were taken using a hand microscope of the Dino-Lite AM7115MZT brand and model. The magnification capacity of the device was set to 250x. An AOB LAB brand and model device was used for microhardness measurements. Samples with dimensions of 20x20x1.5 mm^3 were used for the hardness test. A 500 g load was applied to each sample from 10 different points for 10 seconds to obtain the hardness value. The average of these values obtained was taken and accepted as the Vickers hardness value. A UTS Tribology brand wear analyzer was used for wear tests. The device can perform tests in accordance with ASTM-G99, ASTM-G133, DIN-50324, and other standards related to modules. Analyzes were carried out using the alternative motion test module with the ball on disk method. 100Cr6 stainless steel was used as the ball material and the diameter of the ball was 6 mm. The test was applied under a 2 N load and was carried out at a speed of 2 Hz. In the wear test performed at room temperature, the scar diameter was set as 10 mm, and the analysis was completed with 2500 cycles.

3 Result and discussion

3.1 Morphological analysis

Figure 2 shows the images of Ni-P/ZrO_2 composite coatings prepared with pure nickel and different bath concentrations using an optical hand microscope at 250x magnification. It can be said that the surface morphologies of pure nickel and Ni-P alloy coatings are smoother than other coatings. It is observed that Ni-P/ZrO_2 nanocomposite coatings have a rough surface. It can be said that this situation is due to the distribution of ZrO_2 particles accumulated in the main matrix of the coating. In addition, it is clearly seen on the surface morphology at different current densities. As the current density increases, the structure of the pits formed on the surface grows and disperses. Hosseini et al. [28] investigated the surface morphology of Ni-B/WC composite coatings obtained by electroplating. It was stated that the Ni-B coating has a highly smooth and compact structure, whereas the Ni-B/WC (4 g/L) nanocomposite coating has a porous spherical structure with gaps between clusters, which are known as cauliflower structures. Ünal et al. [29] obtained Ni-B/TiB_2 nanocomposite coatings using the electrodeposition method. In the images obtained with an optical microscope, it was found that the surface morphology of the coatings was generally smooth. However, Ni-B/TiB_2 nanocomposite coating obtained a rougher surface than pure nickel and Ni-B alloy. When the obtained results are compared with the literature, it is revealed that the addition of reinforcement particles to the main matrix affects the surface morphology, and the results are parallel to the literature [30-32].

3.2 Microhardness analysis

In Figure 3, the microhardness results of pure nickel, Ni-P alloy, and Ni-P/ZrO_2 nanocomposite coatings obtained by the electroplating method at different bath concentrations and current densities are given together. A pure nickel sample was also produced for comparison purposes. While pure nickel hardness was measured as 324 HV, Ni-P alloy was measured as 328 and 335 HV. When ZrO2 reinforcement particles were added to Ni-P alloy, it varied between 429 and 456 HV. This situation causes a 40.74% increase compared to pure nickel and a 36.12% increase compared to Ni-P alloy in the hardness analysis of the ZrO_2 reinforcement particle. When compared in terms of current density, the hardness value of the composite coatings produced with a current density of 75 mA/cm^2 is higher than the composite coatings produced with a current density of 50 mA/cm^2. According to these results, increasing the bath concentration amount of ZrO^2 reinforcement particle and increasing the current density significantly affected the hardness results.

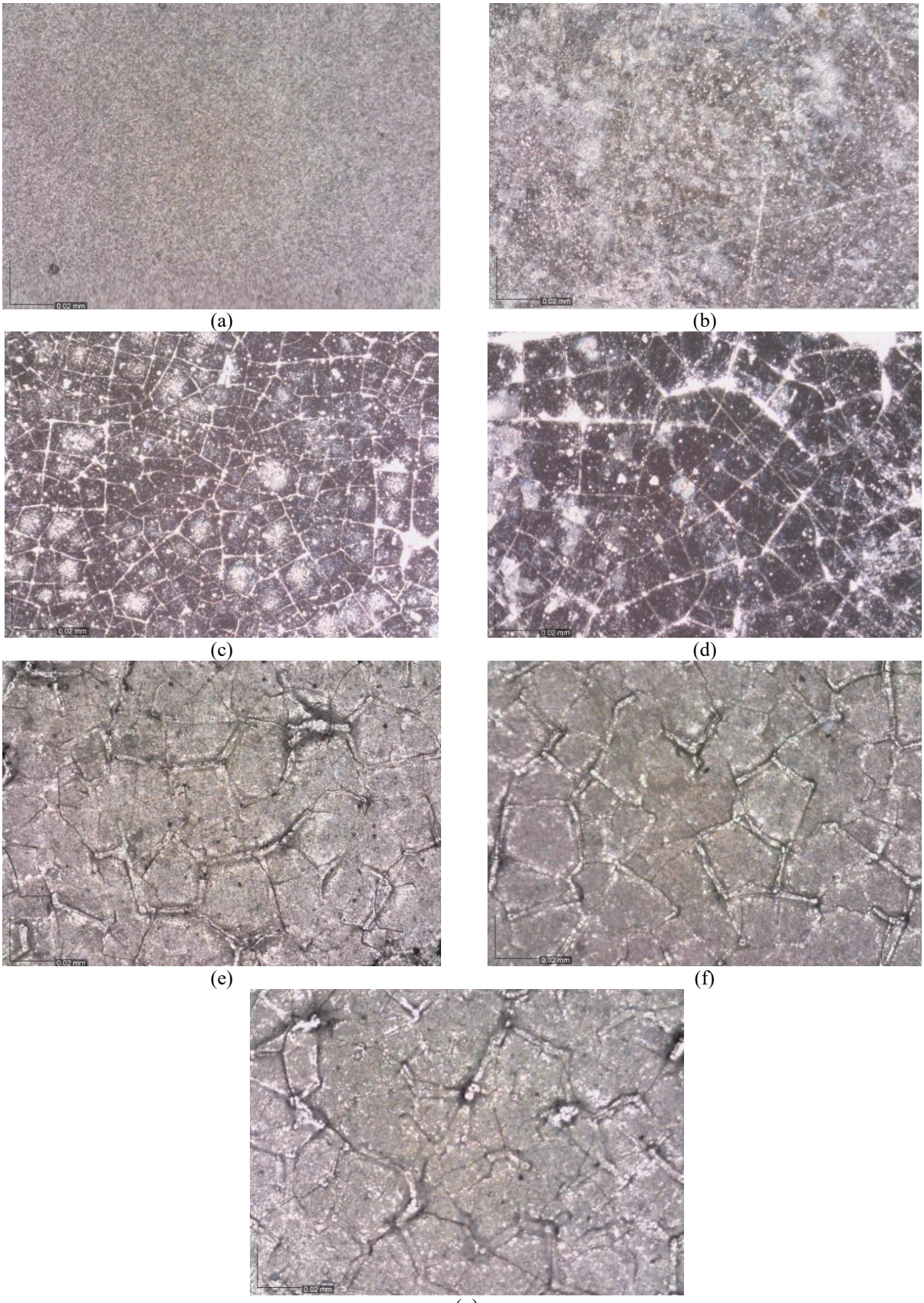

Figure 2. Optical microscope images of coatings (a) Pure nickel, (b) 50NiP0Z, (c) 50NiP10Z, (d) 50NiP20Z, (e) 75NiP0Z, (f) 75NiP10Z, (g) 75NiP20Z

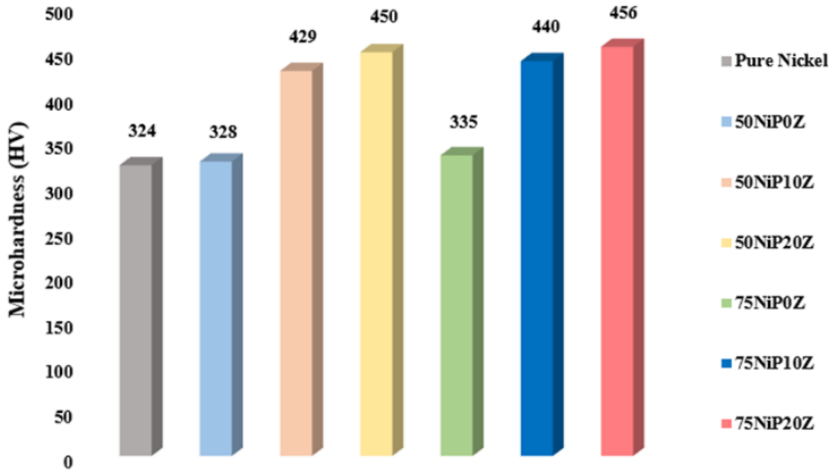

Figure 3. Microhardness values

When the literature is examined, Fayyaz et al. [33] obtained Ni-P/TiC nanocomposite coatings in their studies. Upon examination of the Vickers microhardness and nanoindentation results, the highest recorded microhardness value was found to be 593 HV. It is shown that the hardness increases with the increase in the amount of TiC particles. As seen in the literature, it has been observed that the microhardness of the coating increases up to a certain point by adding the reinforcement particle to the main matrix [34, 35]. When the microhardness of the coatings obtained by the electroplating method is compared in terms of current densities, the coatings produced with 75 mA/cm^2 show very close values to the coatings produced with 50 mA/cm^2, but higher hardness values are obtained. This situation can be considered as accumulating more metal particles by the current sent to the sample. Our study is at the same point as the literature on this subject. Doğan et al. [36] observed an increase in microhardness of the Ni-B alloy coating among the coatings produced when the current density was increased from 1.5 A/dm^2 to 4.5 A/dm^2. Furthermore, the observed increase in hardness can be attributed to the particle's ability to impede dislocation movement, thereby contributing to enhanced resistance against plastic deformation.

3.3 Wear analysis

Wear analyses were performed on pure nickel, Ni-P alloy coating, and Ni-P/ZrO_2 composite coatings. Figures 4 and 5 show friction coefficient graphs and average friction coefficient values.

When the friction coefficient and average friction coefficient graphs are evaluated, it is seen that the nanocomposite coating exhibits lower friction coefficient values than pure nickel. The highest average friction coefficient results were obtained with the pure nickel sample as 0.63 microns, while the lowest average value was obtained with 0.2 microns in the 50NiP20Z coded composite coating. Based on this, while the hardness value increased with the addition of reinforcement nanoparticles to the main matrix, it caused a decrease in the friction coefficient value. In the graph given in Figure 4, it is seen that pure nickel oscillated less than other composite coatings during the test period. While the maximum friction coefficient value of pure nickel was 0.7 microns, the lowest friction coefficient result was seen at 0.65 microns. When the Ni-P alloy coded 50NiP0Z was examined, the maximum friction coefficient value was seen at 0.32 microns levels, while the lowest friction coefficient result was found to be around 0.15 microns. The reason for this is that the friction coefficient value of alloy and composite coatings started low at first and then increased after the sliding layer was deformed. During the wear of the adhered layer, the friction coefficient results first increase and reaches the maximum result, and the friction coefficient value suddenly decreases as the adhered layer breaks.

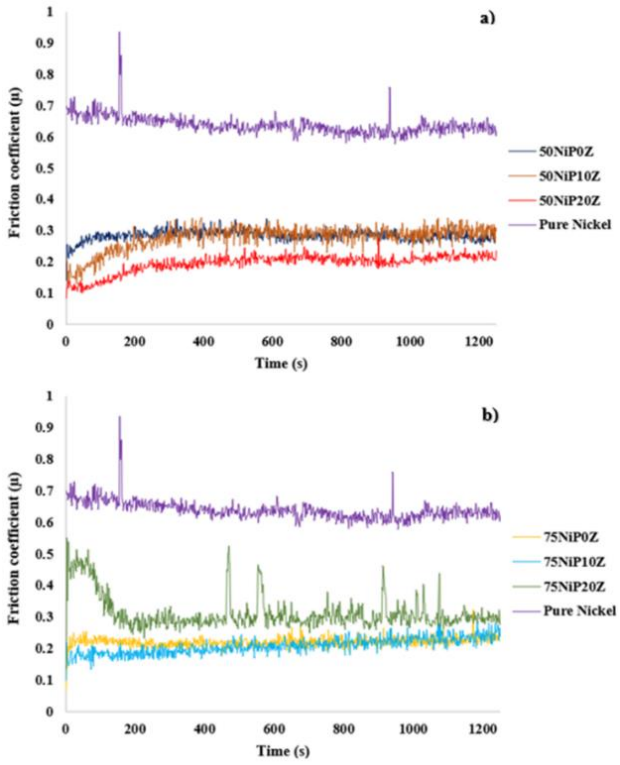

Figure 4. Friction coefficient graph of samples produced with (a) 50 mA/cm^2 current density (b) 75 mA/cm^2 current density

The average friction coefficient values of the coatings obtained with different current densities are given in Figure 5.

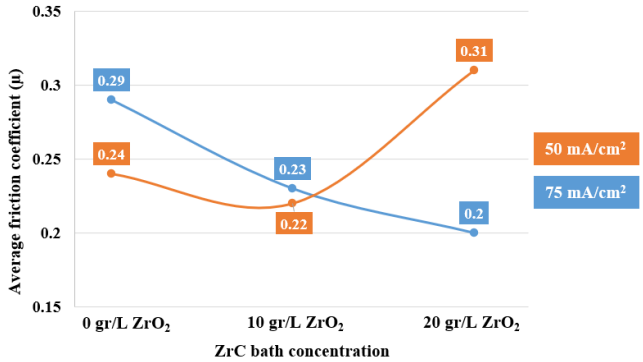

Figure 5. Average friction coefficient results

When the graph is examined, the average friction coefficient values of the coatings obtained with 50 mA/cm^2 current density are generally lower than those of the coatings obtained with 75 mA/cm^2 current density. Only Ni-P/ZrO_2 composite coatings produced at 20 g/L ZrO_2 bath concentration showed a contrasting picture compared to coatings produced at other bath concentrations. The reason for this can be explained by the fact that the ZrO_2 particle does not dive well in the bath. Ünal et al. [29] deposited Ni-B/TiB_2 composite coatings on the surface of AISI 304 steel. The analysis of the wear resistance of the coatings revealed that the coatings demonstrated superior performance compared to pure nickel and the Ni-B alloy coating. While the average friction coefficient result was found to be 0.227 microns for pure nickel, it was found to be 0.187 microns for composite coatings. It is clearly seen that adding TiB_2 reinforcement particles to the main matrix increases the wear resistance. Allahyarzadeh et al. [37] conducted pin-on-disc wear tests to examine the wear performance of the Ni-W coatings performed. The results showed that the obtained coating showed superior wear resistance compared to pure nickel. In addition, the friction coefficient decreased from 0.74 to 0.49 microns with the increase in sliding distance. When looking at the literature, it is seen that adding the abrasion resistance reinforcement particle to the main matrix provides significant abrasion resistance.

4 Conclusion

The optical microscope, microhardness, and wear analysis of Ni-P/ZrO_2 composite coatings obtained by the electrodeposition method using pure nickel, Ni-P alloy, and different bath concentrations and current densities were evaluated comparatively.

- When the surface morphologies of the obtained coatings were examined, coatings with generally smooth surfaces were obtained. With the addition of ZrO_2 nanoparticles to the main matrix, a rougher surface is observed compared to other coatings.
- With the addition of ZrO_2 reinforcement particles, approximately 1.4 times higher microhardness values were found compared to pure nickel and 1.3 times higher than Ni-P alloy. Additionally, an increase in microhardness value was detected with increasing current density.
- When the wear analysis results were examined, composite coatings had an average wear coefficient of 68.25% less than pure nickel and 31.03% less than Ni-P alloy.
- The ZrC reinforcement particle added to the bath concentrate provided superior properties to the composite coatings in terms of surface morphology, hardness and wear performance.

The results obtained from this study show that Ni-P/ZrO_2 composite coatings obtained by the electroplating method are potentially useful in industrial applications and provide enrichment of the literature.

Conflict of interest

No conflict of interest was declared by the authors.

Similarity rate (iThenticate): %14

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