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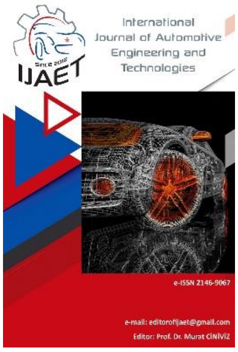

Original Research Article

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Investigation of the effect of bath temperature on Ni-W/Al_2O_3 nanocomposite coatings produced by electrodeposition method

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ABSTRACT

In this study, Ni-W/Al_2O_3 nanocomposite coatings were applied via the direct current electrolysis method on the St-37 steel substrate using a Watts bath. Ni-W alloy coating was obtained by adding sodium tungsten dihydrate as a tungsten source to a Watts-type nickel bath, and then aluminum oxide (Al_2O_3) ceramic nanoparticles were added to this solution to obtain a nanocomposite coating. The effect of different temperatures (25ºC, 60ºC) on Ni-W and Ni-W/Al_2O_3 coatings was investigated and detailed research was carried out on the hardness, surface morphology and wear resistance of the coatings. When the raw information of this study was examined in a logical order, the changes in the hardness of the material and the changes in its mechanical properties showed the effect of temperature on the coating. In general, the surface morphologies of the nanocomposite coatings exhibited a smooth and homogeneous distribution. According to the wear analysis results, with the addition of tungsten element to the main matrix, the average friction coefficient decreased by 36.51% at 25ºC and 46.03% at 60ºC compared to pure nickel. With the addition of Al_2O_3 nanoparticle to Ni-W alloy, the average friction coefficient increased by 12.5% at 25ºC and 2.94% at 60ºC compared to Ni-W alloy. Microhardness results showed that the hardness values increased with the increase in bath temperature. The hardness value of the Ni-W alloy coating obtained at the bath temperature of 60ºC increased by 6.43% compared to the coating obtained at 25ºC. The hardness value of the Ni-W/Al_2O_3 nanocomposite coating obtained at 60ºC increased by 8.72% compared to the coating obtained at 25ºC.

Keywords: Electrodeposition, Nanocomposite, Microhardness, Aluminum oxide, Wear

1. Introduction

The exposure of metallic parts to abrasion and corrosion in industrial environments has led to the need to improve the mechanical properties of materials. There are many solutions for this, but the most effective one is the electroplating process [1]. Electroplating is a process of electrodeposition whereby a dense, uniform and adherent coating is produced on a surface, typically comprising metals or alloys [2].

Nickel-based coatings, one of the metals widely used in electroplating, stand out with their good corrosion properties, wear resistance, and ductility [3]. Nickel-based coatings are frequently preferred in industrial areas such as automotive, aircraft, space, and mining due to the advantages they offer [4]. Nickel coatings are strengthened by metallic particles added to the solution during electrolysis. The coatings formed because of this process are called alloy coatings. Composite coatings are obtained by adding ceramic particles to this alloy coating. Composite coatings have superior mechanical properties to simple metal or metal alloy coatings [5]. Alloy coatings include varieties such as Ni-P [6], Ni-B [7], Ni-Cu [8], Ni-Fe [9], Ni-Ag [10]. In addition, Ni-W coatings attract attention due to the high wear resistance and hardness they offer [11]. Due to these properties, Ni-W coatings stand out in the industry [12]. However, the use of second-phase particles is also important to increase coating efficiency. Silicon carbide (SiC) [13], zirconium oxide (ZrO_2) [14], titanium nitride (TiN) [15], titanium oxide (TiO_2) [16], cerium oxide (CeO_2) [17], titanium carbide (TiC) [18], boron carbide (B_4C) [19], silicon nitride (Si_3N_4) [20], silicon oxide (SiO_2) [21] are widely used nanoparticle types. Another particle widely used in recent years is Al_2O_3 due to its resistance to corrosion and the hardness it adds to the material [22]. Sridhar et al. [23] employed an electrolytic coating process on Ni-W alloys, modifying the temperature parameters. The coating thickness exhibited a 22% increase at the maximum temperature tested. Conversely, an increase in tungsten content in the alloy was observed in the experiment conducted by increasing the current at room temperature, reaching a value of 31%. Fan et al. [24] studied electroplated Ni-W alloy and Ni-W/Al_2O_3 composite coatings. The current density was determined as 5 A/dm^2, temperature as 50ºC, and pH value as 8. As a result, no cracks or damage had lower hardness but better elastic modulus. Sangeetha et al. [25] produced Ni-W/BN nanocomposite coatings on mild steel substrate using direct current and pulse current. The electrolytic process was carried out by adding 2-8 g/L BN nanoparticles to the nickel sulfate bath. BN improved the corrosion resistance and tribological properties of the nanocomposite coatings in the bath. Compared to the coatings produced using pulse current, the corrosion resistance, microhardness, and surface properties of the coatings produced with DC current showed less improvement. Beltowska-Lehman et al. [26] produced Ni-W/ZrO_2 nanocomposite coatings by electrochemical deposition method in a sulfite-citric electrolysis solution. Researchers investigated the bath parameters by changing the bath parameters, such as ZrO_2 particle bath content, current density, hydrodynamic conditions, and frequency. Researchers used approximately 5 g/L nanoparticle content in the coating bath, 600 RPM cathode rotation speed, DC current, and 11 A/dm^2 current density combinations. As a result, the coatings were found to have an approximate hardness value of 8 GPa, wear value of 1.4 10^{-6} mm^3/Nm, and corrosion value of 6 μA/cm^2. Gyawali et al. [27] produced Ni-W/Si_3N_4 nanocomposite coatings with different amounts of tungsten content. Researchers evaluated the wear and microhardness values of nanocomposite coatings by EDS, SEM, XRD and wear test. The researcher's findings showed that the addition of Si_3N_4 and high tungsten addition improved the wear resistance and hardness of the coatings. Also, the coatings with 12.7% W and 2.1% Si_3N_4 content showed the best wear resistance. Liu et al. [28] produced Ni-W/TiN composite coatings using the pulsed current electrolysis method. Researchers investigated the hardness, wear, and microstructure of TiN concentrations prepared using 4, 8, 12, and 16 g/L amounts. Researchers observed that the densest surface structure, the highest microhardness (897.6 HV), and the best wear resistance were observed in the concentration prepared with 8 g/L. They measured the TiN content in this composite as 8.1%. Shakoor et al. [7] Ni–B/Al_2O_3 composite coatings were synthesised via an electroplating process, whereby Al_2O_3 particles were added to a Ni–B matrix. The addition of Al_2O_3 to the Ni–B matrix resulted in a notable enhancement in the crystallinity of the structure. The surface morphology study revealed the formation of a smooth, dense and fine-grained deposit on both the Ni–B and Ni–

B/Al_2O_3 composite coatings. However, the addition of Al_2O_3 particles to Ni–B coatings result in a notable increase in surface roughness. Nanoindentation results demonstrate that the incorporation of Al_2O_3 into the Ni–B matrix significantly enhances the mechanical properties (hardness and elastic modulus) of the composite, which can be attributed to the dispersion hardening of the Ni–B matrix by the Al_2O_3 particles. In this study, the effects of direct current electroplating of Ni-W reinforced with Al_2O_3 were investigated. As a result of this experiment, Ni-W composite coatings reinforced with Al_2O_3 were produced in baths at different temperatures, and the hardness values, wear resistance, and surface quality were investigated. The expected benefits of Al_2O_3 reinforcement particles used in electroplating include extending the service life of the part, increasing impact resistance, and creating a more wear-resistant surface. The primary goal is to improve material quality and investigate the effect of temperature on the coating.

2. Experimental Section

In this experimental study, St-37 steel was used as the cathode, graphene rod as the anode, and a reference electrode. Anode and cathode materials were placed vertically in the bath to be coated, with a distance of 20-30 mm between them, and the surface area of the sample to be coated was set as 4 cm^2. The electrodeposition device arrangement is given in Figure 1.

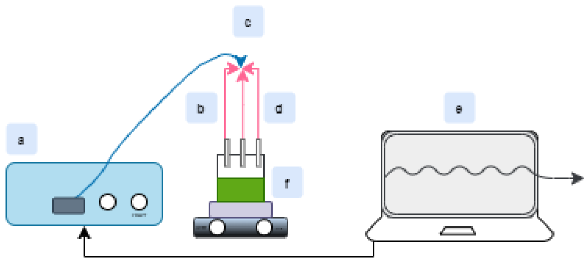

Figure 1. Electrodeposition device arrangement (a) Potentiostat/galvanostat, b) Graphene rod, c) St-37 plate, d) reference electrode, e) computer, f) magnetic stirrer)

Aluminum oxide particles were used with 99% purity and 180 nm size. To make the mixture homogeneous, first, Al_2O_3 nanoparticles and sodium dodecyl sulfate (SDS) were added to pure water and mixed for 30 minutes; then other bath components were added and mixed in an ultrasonic mixer for another 30 minutes. Bath concentrations and parameters are given in Table 1 and Table 2.

Table 1. Bath Components

Bath Components (Chemicals)	**Amount**
Nickel-Sulphate-Hexahydrate	16 g/L
Sodium Tungsten	46 g/L
Ammonium Chloride	26 g/L
Sodium Bromide	15 g/L
SDS	0.5 g/L
Tri Sodium Citrate	142 g/L
Aluminum Oxide (Reinforcement Particle)	5 g/L

Table 2. Operational parameters

Working Conditions	Parameters
Electrolyte Bath pH	9 ± 0.1
Temperature	25°C and 60°C
Bath Shaking Using a Magnetic Stirrer	300 ± 50 rpm
Time (Coating Deposition)	30 min
DC Current Density	50 mA/cm^2

Boric acid or NaOH solution was used to adjust the pH of the electrolytic bath to 9. The temperature of the bath solution was set at 25 ºC and 60 ºC. Before electroplating, St-37 steel material was sanded with 320-600-1200-2000 grit sandpapers, respectively, and then the sample was cleaned in an ultrasonic bath with 20% acetone solution for 30 minutes to remove chemical oils. For the etching process, it was kept in a 10% HCl acid solution for 1 minute. After these processes, the cathode sample was rinsed with pure water and immersed in the electrolyte bath, and the magnetic stirrer was operated at 300 ± 50 rpm to prevent agglomeration of ceramic particles during electrolysis. The current density and pH were kept constant throughout the coating process, and the mechanical properties of the coated samples were analyzed. The number of samples coated, and the bath concentrations of these samples are given in Table 3.

3. Characterization Techniques

The coated samples were examined under the Dino-Lite AM7115MZT model microscope (250x) given in Figure 2.

The homogeneity, smoothness and possible surface defects of the coating were observed.

Table 3. Number of samples coated and their corresponding bath concentrations

Sample (Code Names)	Composition	Temperature (ºC)
N	Pure Nickel	60
NW25	Ni-W	25
NW60	Ni-W	60
NWA25	Ni-W/Al_2O_3	25
NWA60	Ni-W/Al_2O_3	60

Figure 2. Optical microscope testing device

The wear performance of the samples was tested using the ball-disc method and the alternating motion test module. 100Cr6 stainless steel was used as the ball material and the ball diameter was selected as 6 mm. The test was carried out with 2 N load and 2 Hz speed, and the wear test was carried out using the UTS Tribology test device, which can perform tests in accordance with ASTM G133M, ASTM-G99, DIN-50324 standards. The wear test device is shown in Figure 3.

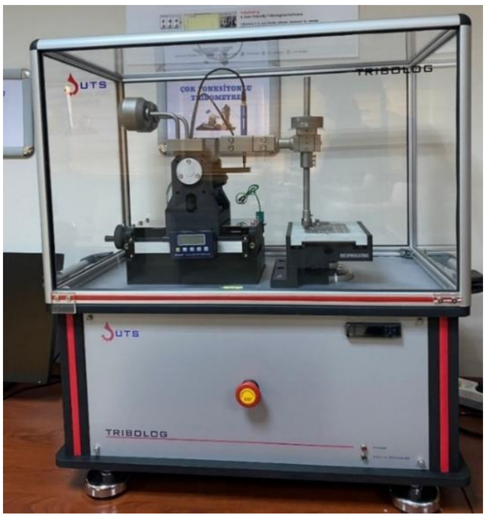

Figure 3. Tribology testing device

For the hardness test, 500 g load was applied to each sample from 10 different points and the hardness value was obtained for 10 seconds. The average of the obtained results was taken and accepted as the Vickers hardness value. Figure 4 demonstrates the Vickers hardness testing device.

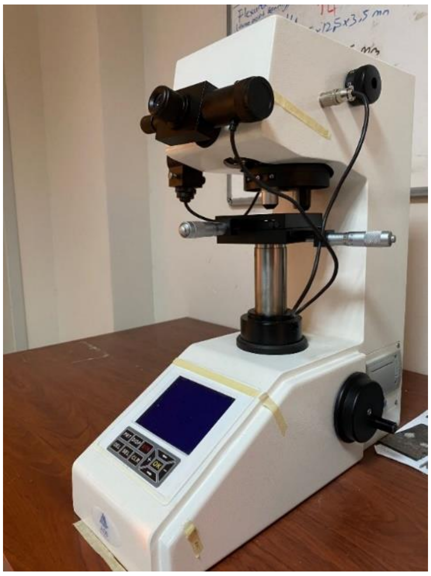

Figure 4. Hardness testing device

4. Results and Discussion

4.1. Surface morphology

Figure 5 shows surface images obtained with a Dino-Lite AM7115MZT model microscope at 250x magnification.

According to the surface morphology findings obtained from the images, the effect of different temperature conditions on the surface properties was observed. While the surface morphology of pure nickel in Figure 4a shows a smooth surface quality, surface defects such as certain roughness and darkening are seen in the surface images obtained from other coatings. In Figure 4b, the surface image of the Ni-W coating produced at 25ºC exhibits a relatively smoother surface structure compared to Figure 4c, while the metallic particles accumulated on the surface show a more homogeneous distribution. This situation showed that the particles added to the Ni main matrix increased the surface roughness. When compared in terms of the effect of Al_2O_3 nanoparticles, it was clearly seen that the surface morphology of Ni-W/Al_2O_3 composite coating performed at 60ºC was more scattered and rougher compared to Ni-W coating performed at 60ºC, thus revealing the effect of added Al_2O_3 particles on the coating.

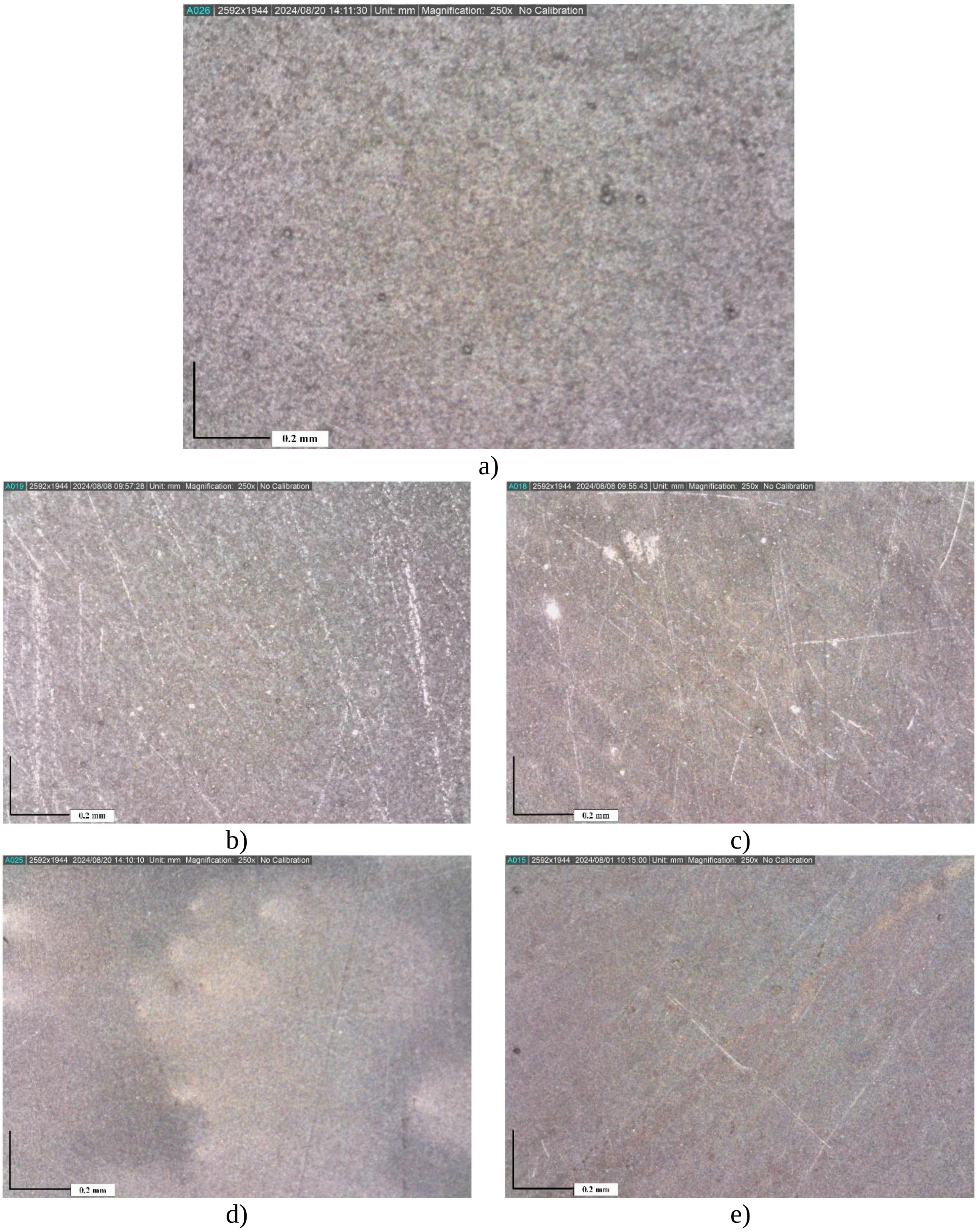

Figure 5. Optical microscope images of the coatings (a) N, b) NiW25, c) NiW60, d) NiWA25, e) NiWA60)

When compared in terms of temperature, a rough surface was seen according to Figure 4d and Figure 4e. The data indicates that elevated temperatures facilitate a more manageable surface during the coating process, thereby reducing the incidence of defects. In general, these findings indicate that coatings at higher temperatures provide better surface quality and minimize surface roughness, and Al_2O_3 additive affects the surface smoothness. All the findings show that the effect of temperature during the coating process plays an important role on the surface morphology. Shakoor et al. [7] demonstrated that the incorporation of Al_2O_3 particles into Ni–B/Al_2O_3 composite coatings resulted in the formation of a smooth, dense, and fine-grained deposit on the surface morphology of both Ni–B and Ni–B/Al_2O_3 composite coatings. Nevertheless, the incorporation of Al_2O_3 particles into Ni–B coatings led to a considerable enhancement in surface roughness.

4.2. Microhardness analysis

The hardness values of Ni-W and Ni-W/Al_2O_3 electroplating obtained at different temperatures are given in Figure 6 for comparison.

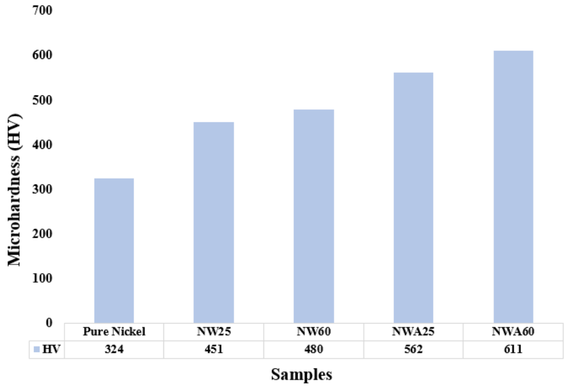

Figure 6. Microhardness values

Microhardness values clearly reveal the effect of temperature on the coated materials. The hardness result of pure nickel was measured as 324 HV. However, tests conducted on Ni-W coating showed that while the hardness value was 451 HV at 25ºC, this value increased to 480 HV when the bath temperature was increased to 60ºC. According to these results, increasing the bath temperature in Ni-W coating leads to an increase in the hardness value. According to these results, a 6.5% increase in the hardness value is observed when the temperature is increased from 25°C to 60°C in Ni-W alloy coating. In addition, a more significant increase in hardness was observed with the increase in temperature in Ni-W/Al_2O_3 nanocomposite coatings. The hardness value of the nanocomposite coating at 25ºC was found to be 562 HV, while when the temperature was increased to 60ºC, the hardness value increased to 611 HV. Accordingly, the hardness values of Ni-W/Al_2O_3 nanocomposite coatings at 60°C increased by 8.72% compared to 25°C. If all the results are compared with pure nickel, with the addition of tungsten element to the pure nickel structure, the microhardness value increased by 39.20% at 25ºC, while a hardness increase of 48.20% was observed at 60ºC. Similarly, when pure nickel is compared with Ni-W/Al_2O_3 composite coatings, a 73.5% increase was observed at 25ºC compared to pure nickel, while an 88.6% increase was observed at 60ºC. These findings show that nanocomposite coatings are more sensitive to bath temperature and hardness data increase more significantly as temperature increases. Temperature sensitivity of Ni-W/Al_2O_3 nanocomposite coatings shows that these materials can provide high performance and durability in industrial applications. As a result, positive effects of increasing bath temperature on the hardness values of coating materials are shown.

4.3. Wear analysis

According to the wear analysis findings, the effects of modifications formed with Al_2O_3 nanoparticles and tungsten at different temperatures on the material performance were remarkable. All these results are given in Figure 7, while the average friction coefficient values are given in Figure 8.

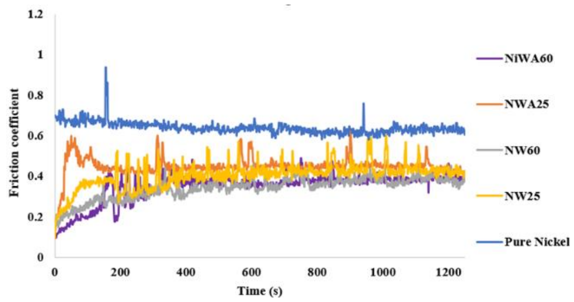

Figure 7. Wear analysis graph of produced samples

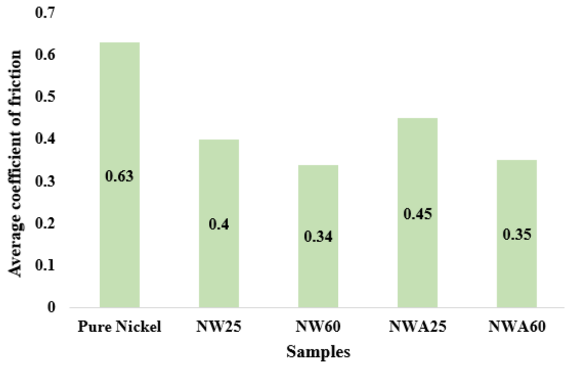

Figure 8. Average Friction Coefficients of Samples

When the results are compared with pure nickel, a significant decrease in the average friction coefficient was achieved with the addition of tungsten to the main matrix. While it was seen that the addition of tungsten reduced the average friction coefficient by 36.51% at 25ºC, these decreases increased to 46.03% at 60ºC. These findings show that tungsten provides a significant improvement in friction and wear and the performance of the material is better improved under high temperature conditions. On the other hand, the addition of Al_2O_3 nanoparticles to the Ni-W alloy increased the average friction coefficient at both temperatures, showing an increase of 12.5% at 25ºC and 2.94% at 60ºC. This increase means that Al_2O_3 nanoparticles do not reduce the average friction coefficient but rather increase it. The integration of Al_2O_3 nanoparticles with the Ni-W alloy caused the average friction coefficient to change to less than 60ºC. In general, while the addition of tungsten increased the average friction coefficient of the material with wear, the addition of Al_2O_3 nanoparticles increased the friction coefficient at different temperature conditions and revealed a different effect profile. In line with these results, it provides important information to understand the effects of different temperature values on material performance and to determine the compatible material according to the usage conditions.

5. Conclusion

The main goal of this study is to compare the mechanical and wear qualities of samples produced at different bath concentrations by the electrodeposition method. Microhardness results, wear test results and optical microscope examinations of pure nickel, Ni-W alloy and Ni-W/Al_2O_3 composite coatings were comparatively analyzed.

- When the surface appearance of composite coatings is evaluated, it is seen that generally smooth surface coatings are produced. In Ni-W coatings, the surface defects of the coating produced at 60ºC are relatively more than those at 25ºC. When looking at Ni-W/Al_2O_3 composite coatings, the surface of the coatings produced at 60ºC is smoother. As a result, increasing temperature has positive results on the surface morphology of composite coatings.
- In nanocomposite coatings, when microhardness values are examined, the effect of temperature on the coating is clearly seen. The increase in temperature resulted in an increase in the hardness value. When we compare NW25, NW60, NWA25 and NWA60 coatings with pure nickel, an increase in hardness was detected. The best hardness value was the Ni-W/Al_2O_3 nanocomposite coating applied at 60ºC.
- When the wear analysis results are examined, it is seen that the average friction coefficients of NW25, NW60, NWA25 and NWA60 coatings decrease compared to the pure nickel coating. Accordingly, it was seen that the average friction coefficient was significantly affected by the addition of W and Al_2O_3 to the Ni structure.

As a result of these findings, it is seen that the most optimum coating is the NWA60 coded coating.

Credit Authorship Contribution Statement

The authors' contribution to the study is equal.

Authors' Conflicts of Interest

There are no financial interests or personal relationships that could have influenced the work presented in this article.

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