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Effect of electroplating temperature on microstructure, corrosion, and wear behavior of Ni-P-W-TiO_2 coating

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Nickel-phosphorus-titanium oxide coating is fabricated on the AISI 304L steel substrate using the electroplating method. Electroplating is performed at temperatures of 55°C, 60°C, and 65°C, and the effect of electroplating temperature on microstructure, corrosion behavior, and wear behavior is investigated. The coatings are characterized using scanning electron microscopy (SEM). In order to investigate corrosion resistance, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) tests are performed in a 3.5% NaCl aqueous solution. A pin-on-disk test is employed to investigate the wear resistance of uncoated and coated samples. Sample micro-hardness is also measured by the Vickers hardness test. The results of potentiodynamic polarization and EIS tests show that the coating created at the temperature of 60°C has the highest corrosion resistance (7058 $\Omega.cm^2$) compared with the samples coated at temperatures of 55°C (2115 $\Omega.cm^2$) and 65°C (2289 $\Omega.cm^2$). Moreover, the results of the wear and micro-hardness test show that the composite coating formed at the temperature of 60°C has the highest wear resistance and micro-hardness (677 Vickers) compared with the samples coated at temperatures of 55°C (411 Vickers) and 65°C (536 Vickers).

Keywords: Ni-P-W-TiO_2 coating, Temperature, Potentiodynamic polarization, Electrochemical impedance spectroscopy, Wear resistance

1 Introduction

Composite coatings are produced by the deposition of oxide particles in a metal matrix from an electrolyte solution or an electrolysis bath. Particle deposition in the metal matrix is of great importance due to excellent mechanical properties such as wear resistance, corrosion resistance, and self-lubrication[1]. Composite coatings have both matrix and secondary phase properties. The most important of these properties are high oxidation resistance, high fatigue resistance, high thermal conductivity, and low thermal expansion coefficient[2]. Electrodeposition is one of the most important methods used to achieve composite coatings. The advantages of this method include precise process control, low energy consumption, the ability to create a coating on parts with complex geometry, and low cost[3].

In general, oxides such as TiO_2, SiO_2, and Al_2O_3, among others are used for high-temperature oxidation resistance as well as hardness and improved wear resistance in nickel matrix[3]. The properties of composite coatings depend on the phases existing in the matrix and the amount and distribution of the sedimentary particles in the coating. These characteristics depend on the process parameters including particle properties (shape, size, concentration, and distribution of particles), the chemical composition of the electrolyte (electrolyte concentration, additives, surfactant type and concentration), and the applied current (direct current, pulse current, and current density)[4]. One of the most important parameters that affects the concentration of deposited particles in the coating, crystal size, corrosion behavior, and mechanical properties of the coating is the electroplating bath temperature[5,6].

In a study by Eom *et al*., the effect of electroplating bath temperature on Co-P surface morphology at temperatures of 60°C, 70°C, and 80°C was assessed. Their results show that as the temperature increase, the number of coating grains decrease, but the grain size was increase[7].

By examining the $Ni\text{-}P\text{-}Al_2O_3$ composite coating, Sheu *et al*. found9 that as the temperature of the bath increased, the particle size first increases and then decreases[8].

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Moreover, by examining the nickel-phosphorus coating, Burchardt showed that the amount of phosphorus in the coating increase with the increase of temperature[9].

According to the literature, no Ni-P-W-TiO_2 coating has been produced on the AISI 304L so far. The presence of TiO_2 is expected to improve the properties of Ni-P-W coatings.

In the present study, the Ni-P-W-TiO_2 composite coating is deposited on the AISI 304L austenitic stainless steel by electroplating. After that, the effect of electroplating temperature on the amount of particles in the coating, microstructure, corrosion behavior, and wear behavior of the coating is investigated. Scanning electron microscopy (SEM) is used to observe the morphology. To investigate the abrasive behavior of these coatings, a pin-on-disk test is performed.

2 Materials and Methods

In this research, AISI 304L stainless steel was used as a substrate for electroplating. The chemical composition of this steel is shown in Table 1. First, the AISI 304L stainless steel sheet was cut using a wire cutter with dimensions of 3 mm × 10 mm × 10 mm. Then a wire with a diameter of 0.7 mm was connected to one side of the sample and the connection was insulated using thermal glue. In the next step, SiC sandpapers with the numbers 60, 120, 200, 500, 800, 1000, and 1200 were used to polish the samples. In order to dehydrate the samples, they were first washed with soap and water and then placed in acetone solution. To activate the surface, the samples were electropolished in a solution containing 10% phosphoric acid for 2 min at current density of 80 $mA.cm^{-2}$. After removing the samples from the electropolish solution, the samples were immediately placed in an electroplating bath for the coating process. The composition of the electroplating bath is given in Table 2. To prepare the bath, the metal salts were first poured into water and dissolved with the help of a full magnetic stirrer. Sodium citrate, sodium hypophosphite, boric acid, and phosphoric acid were added to the bath, respectively. Titanium oxide was eventually added to the bath. After that, the solution was stirred for 6 h to stabilize with the help of a magnetic stirrer. Sodium hydroxide and sulfuric acid were used to adjust the pH. All the experiments were performed in 100 mL of electroplating solution. A nickel plate measuring 2 mm × 20 mm × 20 mm was used as the anode, which was connected to the positive pole. The samples were electroplated at temperatures of 55°C, 60°C, and 65°C. After electroplating, the samples were rinsed twice with distilled water and dried using hot air flow.

Table 1 — Chemical composition of AISI 304L stainless steel based on EDS analysis

Element	C	Mn	Si	P	S	Cr	Ni	Fe
Concentration (Wt.%)	0.031	2.00	0.85	0.04	0.04	19.7	10.8	Bal.

Table 2 — Chemical composition and bath conditions for Ni-P-W-TiO_2 composite coating

Coating mixtures		Electrodeposition parameters/materials	
$NiSO_4.6H_2O$	180.00 g/L	Current density	20 $mA.cm^{-2}$
$NiCl_2.6H_2O$	20.00 g/L	pH	2
Na_2WO_4	20.00 g/L	Electroplating time	45 min
TiO_2	20 g/L	Electroplating temperature	55 °C, 60 °C, 65 °C
H_3BO_3	30.00 g/L	Cathode	AISI 304L stainless steel
H_3PO_4	50 g/L	Anode	Nickel plate
$Na_3C_6H_5O_7$	40 g/L		
$NaPO_2H_2\cdot H_2O$	30 g/L		

The scanning electron microscope (SEM) model of Cam scan MV 2300 equipped with energy dispersive spectroscopy (EDS) analysis was used for morphology and microstructural analysis.

In order to perform electrochemical experiments a common three-electrode cell, including a working electrode (coated or uncoated specimen) and a saturated calomel electrode (SCE), was used as the reference electrode. A platinum electrode was used as an auxiliary electrode. All the experiments were performed in distilled water solution containing NaCl (3.5%) at ambient temperature. Potential control and current measurements were performed using an Autolab PGSTAT302N Potentiostat. Before any testing, each sample was connected to a copper wire and then sealed with epoxy resin. The sample surface was then prepared to be exposed to the solution and the samples were thoroughly rinsed with distilled water and dried with acetone. Moreover, before performing the electrochemical tests, the samples were immersed in the NaCl (3.5%) solution for 1 h in order to establish the equilibrium potential conditions. Electrochemical impedance spectroscopy (EIS) measurements in the corrosion potential, Ecorr, and in the 10^{-2}-10^{-5} frequency range were plotted with excitation amplitude of 5 mV for each sample. Nova and Zview software were used in the impedance tests. Nova software was used as an interface between an impedance device and a computer, and the information obtained by the device was converted into numerical information by this software. To analyze the results obtained from Nova, the information acquired by this software was transferred to Zview software and the data were analyzed using Zview. The impedance spectra were analyzed by Zview software.

In order to investigate the wear resistance of the coatings, the wear test was performed via the pin-on-disk method according to the ASTM G99 standard. The used disk was made of 5100 carbon steel with a hardness of 64 Rockwell and a radius of 200 mm. Prior to the wear test, the loading test began with a force of 2 N and increased by 2 N every 50 m. The experiment continued until a significant reduction in mass was observed in the samples. The wear test was performed at a speed of 0.03 m/s and a distance of 120 m. The amount of weight loss of the samples was weighed with an electronic balance and the results of the friction coefficient were calculated using CDT125 software. In order to investigate the mechanization of wear, the wear path was examined using SEM images.

3 Results and Discussion

3.1 Microstructure of Ni-P-W-TiO_2 coating

Different temperatures were used to study the effect of temperature on the microstructure of Ni-P-W-TiO_2 composite coating. Fig. 1 shows the SEM surface morphology of the electroplated samples at three temperatures of 55°C, 60°C, and 65°C at two magnitudes of 500x and 1500x. The coating formed at the temperature of 55°C is relatively uniform, but there are some cavities and cracks in the surface of the coating (Fig. 1(a)). The coating formed at the temperature of 60°C has no cavities or cracks (Fig. 1(b)). The coating obtained at the temperature of 65°C is non-uniform and discontinuous, and some parts of the substrate remain uncoated (Fig. 1(c)). In addition, the color of the electroplating solution at 65°C changed to a darker color at the primary times of electroplating. After the coating operation at this temperature, some parts of the coating were cracked and detached from the surface of the sample as soon as the temperature of the samples reached the room temperature. At low temperatures (55°C and below), some parts of the sample remained uncoated due to the low rate of reactions. As the temperature rose from 55°C to 60°C, the reaction rate increased and a uniform coating was deposited on the surface. At higher temperatures (65°C), unwanted reactions led to the formation of undesirable hydroxide and oxide compounds. The unwanted reactions that could lead to the production of unwanted and brittle compounds occurred due to the change of the color of the bath solution to a darker color. One of the conditions that can lead to the production of hydroxide compounds is the release of hydrogen in the coating bath[10].

Table 3 shows the chemical composition of the composite coating created at three temperatures of 55°C, 60°C, and 65°C. As the temperature rose from

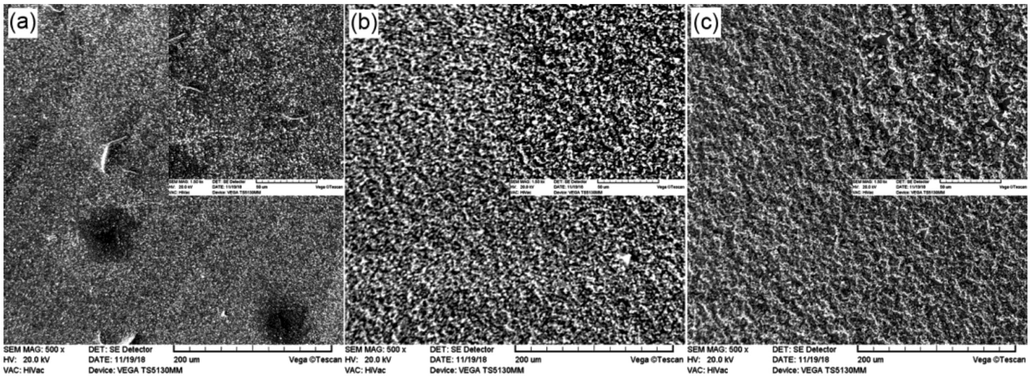

Fig. 1 — SEM surface morphology of the Ni-P-W-TiO_2 coating formed at temperatures of 55°C (a), 60°C (b), and 65°C (c).

Table 3 — Chemical analysis of Ni-P-W-TiO_2 composite coating deposited under different temperatures

temperature (°C)	Ni (wt.%)	P (wt.%)	W (wt.%)	Ti (wt.%)	O (wt.%)	Fe (wt.%)	Cr (wt.%)
55	66.04	9.93	0.62	2.11	3.30	14.13	3.87
60	68.97	7.28	2.08	7.46	14.21	-	-
65	78.06	8.29	1.30	3.89	8.46	-	-

55°C to 60°C, the titanium value increased and then decreased. The presence of iron and chromium elements in the coated samples at temperatures of 55°C and 65°C indicated the presence of cracks and cavities and uncoated spots on the surface, which has been observed in other studies[10].

In general, the coating surface was almost uniform at different temperatures (Fig. 1). In fact, with the increase of temperature from 55°C to 60°C, the uniformity of the coating increased, and then this uniformity decreased with increasing temperature. At high temperatures (65°C), some parts of the surface were uncoated (Fig. 1(c)). This was due to the higher reduction of hydrogen at higher temperatures, which reduced the quality of the surface and affected its mechanical and corrosive properties[11]. Therefore, lower temperatures should be used to achieve high quality coatings[9, 12, 13]. In the process of electrodeposition, the deposition of titanium oxide particles in the coating can lead to an increase in electrochemical potential. The agglomeration of the particles reduces the electric surface of the cathode and also creates new nuclei sites. Moreover, the absorption of particles limits the growth of primary crystals; therefore, both factors are considered for granulation and good surface morphology of composite coatings[14]. Evaluation of the effect of temperature on the amount of TiO_2 particle deposition in the coating revealed that the maximum amount of particle deposition in the coating occurred at 60°C and the titanium deposition value was 7.46 wt.% (Table 3). The reason for this behavior can be explained by the fact that as the temperature increases, the movement speed of ions towards the cathode increases, and therefore the rate of Ni deposition increases. In addition, with the increase of temperature, the movement of TiO_2 particles increases due to increased turbulence[14]. However, excessive increase of temperature results in the release of hydrogen on the cathode surface, which leads to the formation of hydrogen bubbles. The formed bubbles prevent the precipitation of nickel particles and TiO_2 particles on the cathode surface and reduce the amount of precipitation[15]. The coating generated at 55°C contained cavities and cracks. At low temperatures, the electrophoretic force that moves the particles to the cathode is low, resulting in lower particle deposition. When the temperature rises excessively (65°C), the particle motion increases to such an extent that the particles are separated from the surface before the second stage of adsorption due to high activity[16]. At low temperatures, the nickel ions transfer faster than the transported TiO_2 particles through mechanical turbulence, thus reducing the deposition of the TiO_2 particles in the coating[17, 18].

The created cracks on the surface of the sample coated at 55°C can be due to residual stresses. Electrolytic coatings are usually associated with residual stresses. Residual stresses have different origins, such as non-compliance with crystallography and differences in the coefficient of thermal expansion between the substrate and the coating layer. These stresses may be tensile or compressive depending on the matrix of the coating and the parameters of the process. Tensile stresses reduce the fatigue life, efficiency, and spallation in corrosive environments[15]. Factors such as the release of hydrogen at the cathode site and the subsequent increase in pH near the cathode surface result in the entrapment of OH^- or O^{2-} as $Ni(OH)_2$ or NiO compositions, leading to the increase of stress[19, 20].

3.2 Corrosion behavior of Ni-P-W-TiO_2 coating

Fig. 2 demonstrates the graphs of potentiation dynamics polarization test for the Ni-P-W-TiO_2 composite coating formed at temperatures of 55°C, 60°C, and 65°C. Table 4 shows the obtained information from the polarization curve of Ni-P-W-TiO_2 composite coatings. According to Fig. 2 and Table 4, among the three samples coated at temperatures of 55°C, 60°C, and 65°C, the sample coated with the Ni-P-W-TiO_2 composite at the temperature of 60°C had higher corrosion resistance and nobler potential, lower corrosion current density, and lower corrosion rate than the sample coated at temperatures of 55°C and 65°C. According to Table 3, the amount of titanium oxide was the highest in the

Table 4 — Electrochemical parameters obtained from the polarization curve of Ni-P-W-TiO_2 composite coating formed at different temperatures

Temperature (°C)	E_{corr} (V)	i_{corr} ($\mu A.cm^{-2}$)	corrosion rate (mm/year)	polarization resistance ($\Omega.cm^2$)	βa (V/dec)	βc (V/dec)
55	-0.1787	3.55	0.03980	7783.7	0.12329	0.085268
60	-0.1887	1.21	0.014059	21536	0.13913	0.073453
65	-0.2233	1.88	0.021789	13897	0.12525	0.074761

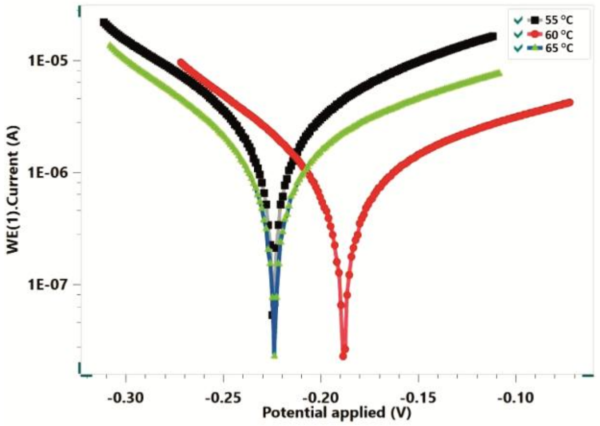

Fig. 2 — Potentiodynamic polarization curves of the Ni-P-W-TiO_2 composite coating formed at different temperatures in 3.5% NaCl solution.

coating layer formed at 60°C. The higher amount of ceramic particles led to the decrease of the current through the surface, thus improving the corrosion resistance.

Ceramic particles fill the microscopic cracks and holes of the coating. These particles act as barriers and ineffective sites for the initiation and development of corrosion, thus improving the corrosion resistance of the coating[21].

To investigate the anti-corrosion properties of composite coatings obtained at temperatures of 55°C, 60°C, and 65°C, the EIS test was used in 3.5% NaCl solution. Fig. 3 shows the Nyquist curves of the composite coatings produced at 55°C, 60°C, and 65°C. Figures 4(a and b) also show the bode and bode-phase diagrams for the tested samples, respectively. All the curves are drawn in the frequency range of 0.01 Hz to 100 kHz. The results of fitting the equivalent circuits using the Zview software are given in Table 5. As observed, the impedance results are well consistent with the Tafel polarization results. In this table, R_S is the solution resistance, R_P is the resistance of the coating against the penetration of the solution through the defects and pores, and CPE is the constant phase element of the double layer, which is used instead of the simple capacitor. In order to achieve a satisfactory simulation of the impedance data of composite coating, CPE replaced a simple capacitor. The most important justification for this matter is the presence of microscopic roughness on the coatings, leading to the non-uniform distribution of the solution resistance and the capacity of the double layer[22].

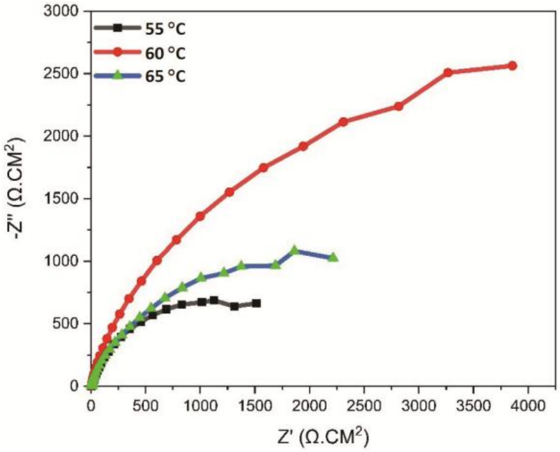

Fig. 3 — Nyquist diagram of the Ni-P-W-TiO2 composite coating formed at different temperatures in 3.5% NaCl solution.

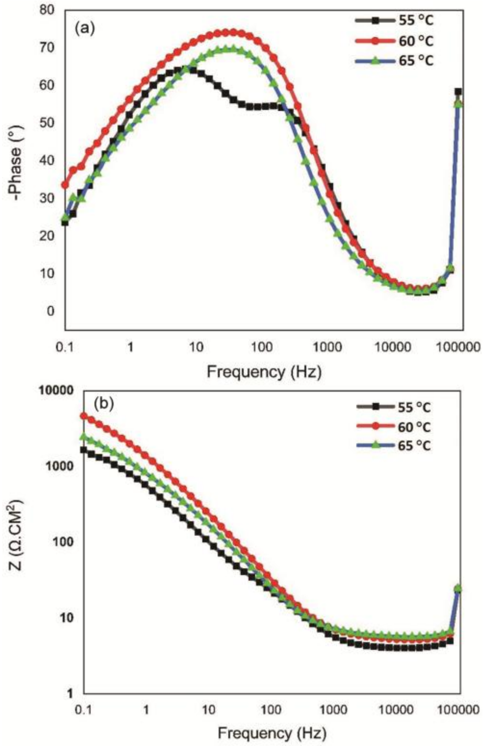

Fig. 4 — Curves of (a) bode, and (b) bode phase for the Ni-P-W-TiO_2 composite coating formed at different temperatures in 3.5% NaCl solution after the wear test.

As can be seen in Fig. 4(a), bode curves have almost two distinct peaks for coated specimens; therefore, it can be said that the corrosion process of the coated specimens includes two constant times. The existence of two rings in the Nyquist curves also proves this matter. The first constant time is related to high frequencies and the interface of the coating/electrolyte, and the second constant time is related to low frequencies and indicates the conditions of the substrate/electrolyte interface[23].

The presence of peaks at high frequencies in the bode curves of the coated specimen at the temperature

Table 5 — Electrochemical parameters obtained from electrochemical impedance for samples coated at different temperatures

Temperature (°C)	R_S (Ω.cm^2)	R_c (Ω.cm^2)	CPE1-T (F.cm^{-2})	CPE1-P (F.cm^{-2})
55	3.849	2115	0.00039098	0.74882
60	4.922	7058	0.00017377	0.75175
60	5.587	2289	0.00021349	0.81843

of 60°C confirms that the coated specimen effectively performed its protective role. The impedance module can be measured using bode curves. The higher impedance module at lower frequencies indicates the better protective properties of the coating. Since the impedance module for the coated specimen at 60°C had a higher value than other coatings, it can be concluded that electroplating at this temperature results in more resistance in corrosive environments.

Moreover, it is clearly observed that the coated specimen at 60°C is more resistant in comparison with coatings formed at lower or higher temperatures. The EIS obtained from the coatings formed at lower or higher temperatures shows circles with lower diameters. In fact, the diameter of the semicircles determines the anti-corrosion properties of the coatings, and a larger diameter ensures better corrosion resistance. Therefore, it is apparent that the composite coating formed at 60°C had the highest corrosion resistance and better anti-corrosion properties.

Higher CPE-T values indicate more porosity in the coating. The coating formed at 55°C had higher CPE-T than other coatings; therefore, it had more porosity and the penetration of electrolyte to the coating and substrate could occur easier. According to Table 5, the coated sample at 60°C had the lowest CPE-T value compared with other coatings; therefore, it had the lowest amount of porosity.

3.3 Wear resistance of Ni-P-W-TiO_2 coating

The micro-hardness of the Ni-P-W-TiO_2 composite coating was measured based on the described experimental procedure. With the increase of temperature from 55°C (411 Vickers) to 60°C (677 Vickers), the value of micro-hardness increased and at the temperature of 60°C, the maximum hardness was obtained. Then with the increase of temperature to 65°C (536 Vickers), the hardness decreased. The reason for this behavior is the increase of deposited titanium oxide at 60°C and then the decrease in the amount of TiO_2 with the increase of temperature. Increasing the amount of titanium oxide increases the hardness[23].

The weight loss changes after the wear test for the uncoated and coated samples at different temperatures were also measured. The weight loss of the coated sample at 55°C was 77 g^{-4}, and as the temperature rose to 60°C, the weight loss decreased. The coated specimen at 60°C had the lowest weight loss (4 g^{-4}) and with the increase of temperature to 65°C the weight loss of the specimens increased (28 g^{-4}). The wear test was performed at a force of 2 N and a distance of 120 m. The TiO_2 particles can be removed from the surface during wear and reduce the contact surface between the pins and the composite coating. These particles play the role of solid lubricants[15]. As a result, the wear resistance of the coating decreased with decreasing TiO_2. On the other hand, with the increase of temperature (65°C), the hardness decreased, which led to the decrease of wear resistance. The highest weight loss was related to the 304 steel and the lowest weight loss was related to the coated sample at 60°C[24]. Moreover, according to Fig. 1, the lowest level of non-uniformity was related to the coated sample at 60°C. Comparison of the obtained information from the wear test of the coatings created at different temperatures showed that the increase of TiO_2 particles in the matrix of Ni-P-W coating improved the wear resistance of the composite coating. There is a direct relationship between hardness and wear resistance; therefore, wear resistance increases with the percentage of oxide deposition. This behavior has also been observed in Ni-P-Si_3N_4, Ni-P-SiC, and Ni-P-diamond composite coatings[24-26].

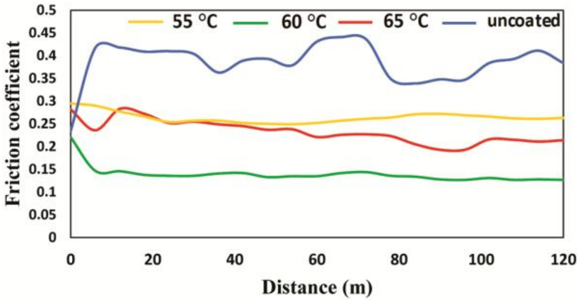

Fig. 5 — Friction coefficient as a function of wear distance for uncoated and coated samples at different temperatures.

One of the factors that affect the wear resistance is the coefficient of friction[27]. Fig. 5 demonstrates the effect of coating temperature on the friction coefficient of the coated samples. The highest friction

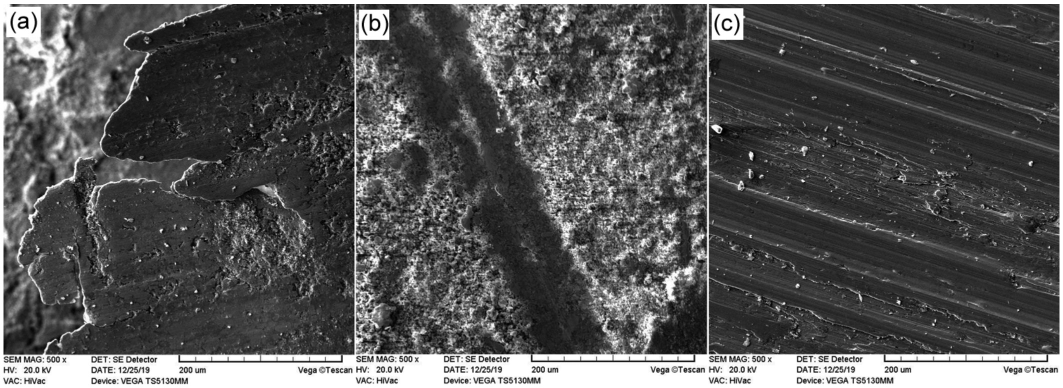

Fig. 6 — SEM surface morphology after wear test of the Ni-P-W-TiO_2 coating formed at different temperatures of (a) 55°C, (b) 60°C, and (c) 65°C.

coefficient was related to the uncoated 304 steel, and all the composite coating samples had a much lower friction coefficient than the 304 steel substrate. The Ni-P-W-TiO_2 composite coating created at 60°C showed the lowest friction coefficient. At lower (55°C) and higher (65°C) temperatures, due to the reduction in the amount of TiO_2 particles, the solid self-lubrication decreased and the friction coefficient increased[28].

Figure 6 shows the SEM image of the Ni-P-W-TiO_2 composite coatings formed at temperatures of 55°C (Fig 6(a)), 60°C (Fig 6(b)), and 65°C (Fig 6(c)). The sample coated at 60°C had a smooth-looking wear path, and the wear path width was the lowest in this sample. In this case, a relative wear was created on the coating, which was due to the higher amount of TiO_2 particles in the coating than the coatings created at temperatures of 55°C and 65°C. The wear mechanism for the sample electroplated at 55°C was delamination. The produced grooves at the surface of the sample coated at 55°C were deeper and wider than the other samples. The width of the wear path decreased with the increase of temperature. As the temperature increased to 60°C, the wear resistance increased due to the increase of TiO_2 particles in the coating. As the temperature rose from 60°C to 65°C, severe wears occurred with the mechanisms of adhesion and delamination. As a result, the effects of plastic deformation and wear on the wear path were noticeable.

TiO_2 particles improved the tribological properties and hardness of the Ni-P-W-TiO_2 composite coating by the distributive hardening mechanism. Hard TiO_2 particles can prevent the movement of metal grain boundaries and prevent grain growth. The higher the amount of hard particles and the more the uniform distribution are, the larger the number of locked dislocations will be. Therefore, due to the increase in the surface area of TiO_2, the interfaces between the phases increased, the grain boundary mobility decreased, and as a result, the wear resistance increased[26]. The coated sample at 60°C had the highest wear resistance compared with the other samples.

4 Conclusion

The Ni-PW-TiO_2 composite coating is fabricated on the AISI 304L stainless steel through the electroplating method, and the effect of different temperatures (55°C, 60°C, and 65°C) on the microstructure, corrosion behavior, and mechanical properties is investigated and the following results are obtained:

- With the increase of temperature to 60°C, the precipitation of ceramic particles and tungsten in the coating increase.
- The coating created at the temperature of 60°C shows a uniform surface without cracks and cavities.
- The coating created at the temperature of 60°C shows the lowest corrosion current density and the highest corrosion resistance. In addition, the increase of titanium oxide particles lead to the decrease of porosity, which reduces the penetration of electrolytes into the coating and increases the corrosion resistance of the coating.
- With the increase of the temperature to 60°C, the hardness of the composite coating increases.
- The coating created at the temperature of 60°C shows the lowest weight loss and the highest wear resistance. The higher presence of titanium oxide in the coating and the reduction of the friction

coefficient leads to the increase of hardness and wear resistance.

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