

RESEARCH PAPER

TiO₂ nano-particle effect on the chemical and physical properties of Ni-P-TiO₂ nanocomposite electroless coatings

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ABSTRACT

TiO₂ nano-particles were used in electroless plating bath to obtain Ni/P/nano-composite coatings. The coatings were heat treated at 200, 400, 600 and 700 °C and their chemical and physical properties were investigated and it was found that 400 °C was the optimum temperature for the heat treatment of the coatings. The micro-hardness test of coatings showed that the composite coatings, which contain TiO₂ nano-particles, exhibit better properties of microhardness. X-ray diffraction (XRD) analysis indicated that at 400 °C, Ni₃P phase is formed, and when the heating temperature is 600 °C the presence of TiO₂ particles is seen. We have used scanning Electron Microscopy (SEM) to measure the surface morphology of the composite and plane deposits. Weight loss measurement, Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization Spectroscopy were utilized to study the corrosion resistance of coatings in 3.5 %wt NaCl solution. Corrosion resistance experiments indicated that presence of TiO₂ nano particles in the electroless coatings matrix improved the corrosion resistance of the coatings. Heat-treatment improved the corrosion resistance of the coatings up to 400 °C but heating above 400 °C caused a decrease in corrosion resistance. Wear behavior of the samples indicated that presence of TiO₂ particles improve the wear resistance.

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INTRODUCTION

Electroless Nickel (EN) coatings have been widely used in aerospace, mechanical and chemical industries because of their surface hardness, corrosion, oxidation, and wear resistance [1-9].

In electroless plating reaction, nickel ions are catalytically reduced on active substrates without using electrical energy, therefore, the needed electrons for reduction reaction is provided by co-chemical reducing agent [10]. One of the main co-chemical reducing agents is phosphorous and the resulting EN is called Nickel-phosphorous (Ni/P) plating and the alloys has excellent mechanical,

electrical, magnetic and anticorrosive properties.

For electroless process, one of the key factors is the content and the property of the bath solutions. The effect of these conditions on various substrates which have a significant influence on the deposition rate, structure of chemical composition and the quality of the coated film have been studied extensively in the past [11-19]. For example, Gan et.al. [20], have studied the effect of bath composition, pH and temperature on the electroless plating of Cu-Ni-P alloy on polyethylene terephthalate (PET). In other work, effect of molybdate conversion film as

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pretreatment layer on the nickel plating on Mg-8Li was investigated and found that the molybdate layer reduces the potential difference between coating substrate and also increases the corrosion resistance of the alloy [21]. In addition, the effect of pH and the amount of Cu on the morphology, hardness and the amount of alloying elements have been studied previously and shown that the increasing the pH, not only reduces the amount of P in the final deposited composition also, increases the coating rate [22].

With recent advances in nanoparticles technology and their applications in different fields [23-30], also, they have been used as an additive in electroless Ni/P plating. Composite coating is prepared by adding solid particles such as alumina, titanium oxide, silicon carbide, silicon nitride, carbon nanotube into electroless coating solution [31]. These coatings show better physico-mechanical properties compared with plane Ni/P electroless coatings. Nickel / Phosphorous/Carbon Nanotube electroless composite coatings showed that these coating have great microhardness, higher wear resistance and lower friction coefficient compared with Ni, Ni/SiC and Ni/Graphite composite coatings [32]. Electrochemical and metallurgical properties of Ni/P/ZnO nanocomposite coatings were improved due to the preserve of ZnO nanoparticles in Ni/P matrix [33]. Similar results are reported for Al₂O₃ [34-36] Si₃N₄ [37], WC [38], Gold [39], TiN [40], Ca_{0.9}Yb_{0.1}MnO₃ [41] and Ni [42] nano particles. In addition, more recently, the effect of carbon

fibers on Ni/P plating's is investigated [43] and to prevent the crack propagation in the deposited layers, the multilayered depositions have been proposed [44].

The aim of the present work is to obtain electroless Ni/P and Ni/P/TiO₂ nano composite coatings and investigate the effect of TiO₂ nano particles and their heat treatment on the corrosion resistance of the coatings.

MATERIALS AND METHODS

An electroless plating bath was used to obtain nickel-phosphorous and composite coatings. Bath composition and optimal conditions are listed in Table 1. The pH of the bath was adjusted with NaOH (10%wt) and the temperature of solution was controlled. CK75-Carbon Steel (contain 0.75% carbon, 20×15×1 mm dimension) was used as substrate. The substrate cleaned with 10%wt NaOH solution, washed and rinsed with distilled water and then dried and placed vertically in to the bath.

Composite coatings were obtained with by adding various contents of TiO₂ nano particles to the bath. Before starting the plating process, TiO₂ particles dispersed in the bath using ultrasonic device (Eurosonic- 4D with frequencies 50/60 Hz) for about 30 min.

During deposition, the electrolyte was stirred at a constant speed of 100 r/min with PTFE magnet stirrer. Hardness measurements were carried out using Shimadzu hardness tester, with Vickers indenter, at a load of 50 gr and duration of 15 s.

Table 1. Bath composition and operating conditions

Bath composition and operating conditions	Concentration
NiSO ₄ .6H ₂ O	25 (gr/L)
NaH ₂ PO ₂ .XH ₂ O	18 (gr/L)
Na ₃ C ₆ H ₅ O ₇	18(gr/L)
CH ₃ COONa	20(gr/L)
TiO ₂ (nano-powder)	5/10/15/20/25/30(gr/L)
(NH ₂) ₂ CS	1 ppm
pH	5.5
Temperature	88±1 (°C)

Three readings were made on each specimen and the values were then averaged. The structure of coatings was analyzed using X-Ray diffractometer (Philips Expert) with Cu-Ka radiation, $\lambda = 1.54 \text{ \AA}$ in 40 kV and 40 mA.

Surface morphology was analyzed by Scanning Electron Microscope (SEM, Philips-XL/30). The chemical composition was determined by Energy-dispersive X-ray spectroscopy (EDX) measurement on instrument SEM. To evaluate the corrosion resistance of the coatings with and without heat-treatment, weight loss measurements were carried out. Electroless deposited steel samples of size 10×10×1 mm washed and rinsed with distilled water, weighted and placed vertically for 100 hr at 30 °C in 50 cc beaker containing a corrosive aqueous solution of 3.5 %wt NaCl. Solution was not stirred during this period. After 100 hr immersion, samples taken out from solution, cleaned with water and then dried and weighted again.

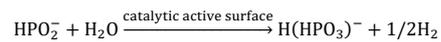
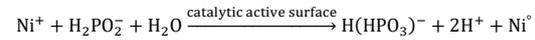
The electrochemical Potentiostat/Galvanostat devise (Auto-Lab 20A) was used to obtain polarization curves. The polarization curves were also obtained from Electrochemical Impedance Spectroscopy (EIS) measurements. The cell assembly consisted of Saturated Calomel Electrode (SCE) as reference electrode, platinum foil as counter electrode and the coated sample as working electrode. Each sample was first immersed in corrosive solution for 30 min in order to establish the free corrosion potential and the scanning rate was 0.01 volt.s⁻¹. In EIS

measurements the frequency range was 100 mHz to 100 KHz. In all corrosion tests an aqueous of NaCl 3.5%wt was used. The solution was open to air and the area of 1 cm² of samples was immersed in the corrosion solution.

The wear test was conducted using pin-on-disc wear tester under dry condition at 25 °C. The test parameters were Load 5N, Velocity 0.1m/s.

RESULTS AND DISCUSSION

Many different mechanisms have proposed for the reduction of Nickel ion by hypophosphite. In one the most accepted electrochemical mechanisms [ref], it has been proposed that hypophosphite ion is catalytically oxidized and nickel and hydrogen ions are reduced at the catalytic surface. Therefore, the main reactions will be expressed by the following equations:



Our findings show a direct dependency between the content of incorporated TiO₂ particles into the nickel-phosphorous matrix and the microhardness of coatings. An increase in the microhardness obtained using TiO₂ nano-particles. These results depicted in the Fig. 1 in a bar chart form. We obtained the microhardness values of as-deposited and heat-treated Ni-P and Ni/P/TiO₂ coatings in the optimum heating temperature (400

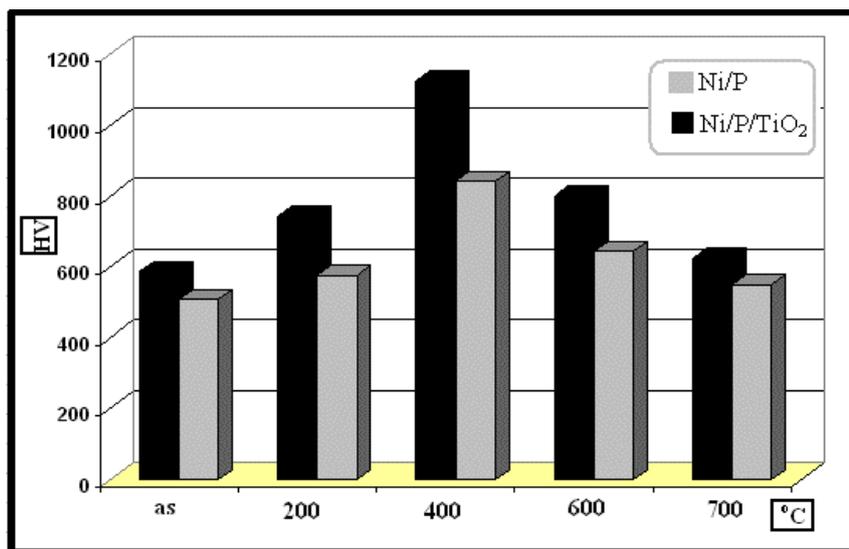


Fig. 1. Microhardness values of plain and composite coatings.

°C) as 508, 841, 589 and 1124 HV, respectively. It is clear from this figure that the microhardness of composite coatings comparing to that of the simple Ni-P coatings is higher. In addition, the heat treatment of the samples shows an improvement in the microhardness of deposits in the cases of with and without nano-particles. Our data also shows that maximum of hardness have achieved at heat treatment when the temperature is about 400 °C. It has been reported that Ni₃P phase have formed between 250 °C and 400 °C that causes the increase in the hardness of the coatings [45, 46]. However, above 400 °C the microhardness value of

both plane and composite coatings decreases and this is because of the oxidation of coating surface at higher temperatures.

The X-ray diffraction (XRD) pattern of Ni/P and Ni/P/TiO₂ composite coating and heated samples (both in 400 °C) are presented in Fig. 2. The XRD analyses for plane Ni/P coating present a broad peak at diffraction angle 44.8°. This has been confirmed by other works and the broad diffraction pattern is related to the (111) plane of a face centered phase of Nickel [45]. This figure shows a peak at 41.77° corresponds to Ni₃P (321) (JCPDS No. 65-2778), peak at 44.68° corresponds

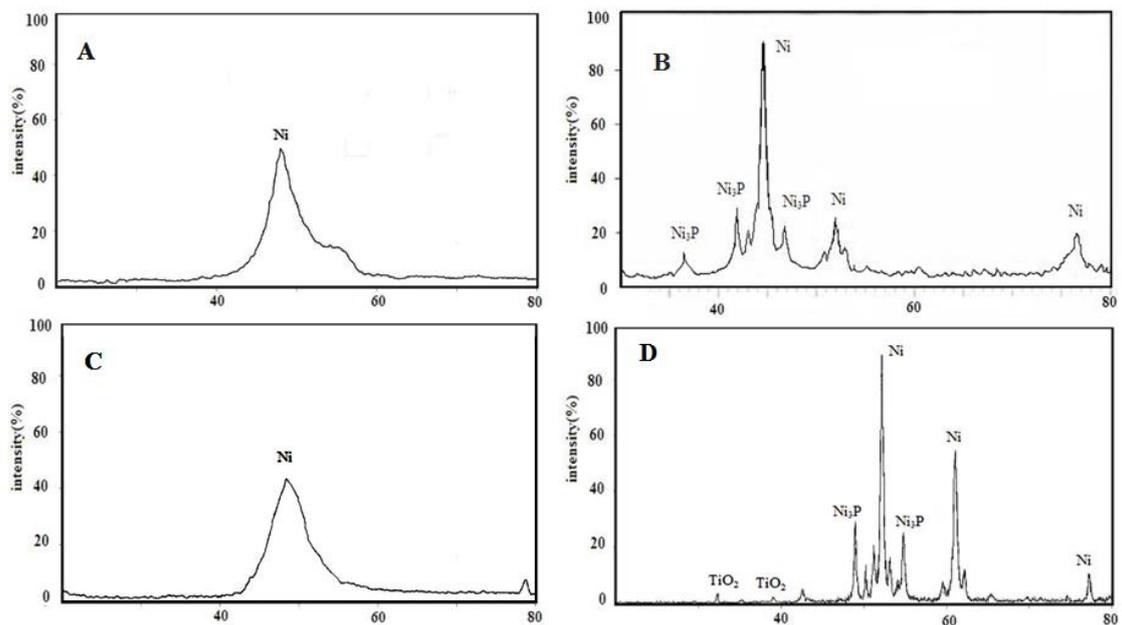


Fig. 2. The XRD pattern of coatings (a: as deposited and, b: Heated Ni/P coating (at 400 °C), c: as deposited and, d: Heated Ni/P/TiO₂ coating (at 400 °C)).

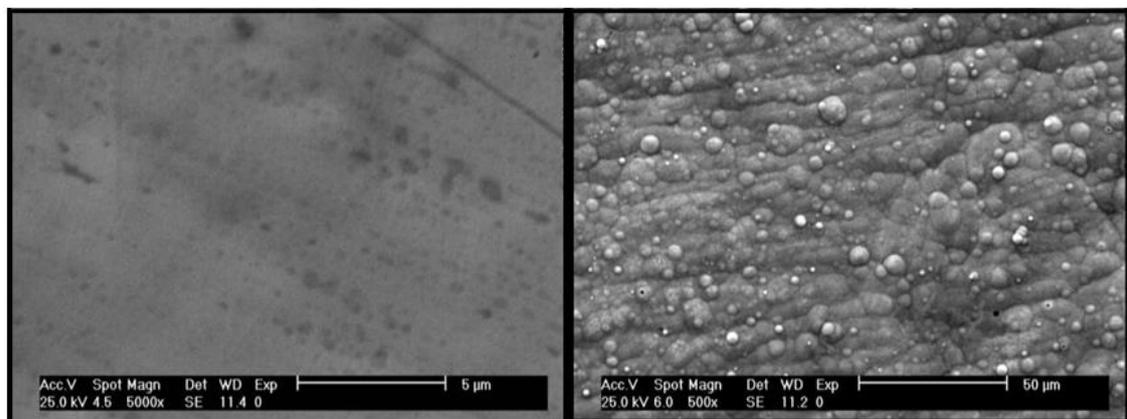


Fig. 3. SEM images for plain (left) and composite coatings (right).

to Ni (111) (JCPDS No. 03-1051), peak at 46.57° corresponds to Ni₃P (141) (JCPDS No. 34-0501), peak at 51.93° corresponds to Ni₃P (150) (JCPDS No. 89-2743) and Ni (220) (JCPDS No. 88-2326) peak appears at 78.21°. It is clear that at 400 °C Ni₃P phase are formed, also presence of TiO₂ particles in the coating is seen, when the heating temperature is 600 °C.

Fig. 3 shows the surface morphology of coatings obtained using SEM. It is clear that co-deposition of nano-particles changes surface morphology of Ni-P coating and smooth surface of plane Ni-P changed to none-smooth and nodular surface. Also, the uniform distribution of TiO₂ nano-particles in the composite coating can be seen in the X-Ray Map of Titanium atoms in composite coating that obtained using wavelength-dispersive spectroscopy (WDS) analyzer on instrument SEM, shown in Fig. 4.

Weight loss measurements for as deposited and heat treated (H.T), at 400 °C, plane and composite coatings after 100 hr immersion in NaCl bath are shown in Table 2. This experiment shows that presence of TiO₂ nano-particles has improved the corrosion resistance of coatings. Also heat-

treated coatings have better corrosion resistance compared with as-deposited ones.

Nyquist plots provide useful information about the corrosion behavior of coated substrates. In nyquist plots the opposite of imaginary part of impedance (Z_{im}) is plotted against real part (Z_{re}). The EIS data of coatings were displayed as a nyquist plot in Fig. 5. Differences between plain and composite coatings and also between before and after the heating processes are clear. In nyquist plot curve A, B, C and D show corrosion resistance of Ni-P Coating, Ni-P coating heat treated in 400, Ni-P-TiO₂ coating, Ni-P-TiO₂ coating heat treated at 400 respectively. The results show that the impedance value of composite coating and composite heat-treated coating are higher than plane one and these results are quite consistent with those obtained from polarization tests.

Fig. 6 shows the results obtained from polarization studies for heated and as-deposited Ni/P coating. Corrosion current densities (i_{corr}) for as-deposited and annealed coatings at 200, 400, 600 and 700 °C were 5.4, 3.2, 2.1, 4.6 and 7.6 $\mu\text{A}\cdot\text{cm}^{-2}$ respectively. Heating at 200 and 400 °C improve the corrosion resistance of plain coatings

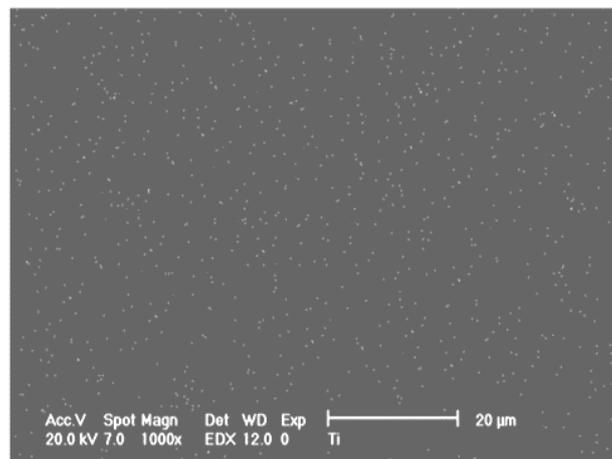


Fig. 4. WDS analyses for Titanium atoms in the matrix of composite coating.

Table 2. Weight loss experiment results (mg/100hr) in 3.5% NaCl solution at 30 oC

Ni/P		Ni/P/ TiO ₂	
As deposited	Heat treated	As deposited	Heat treated
5.5	6.1	4.7	3.9

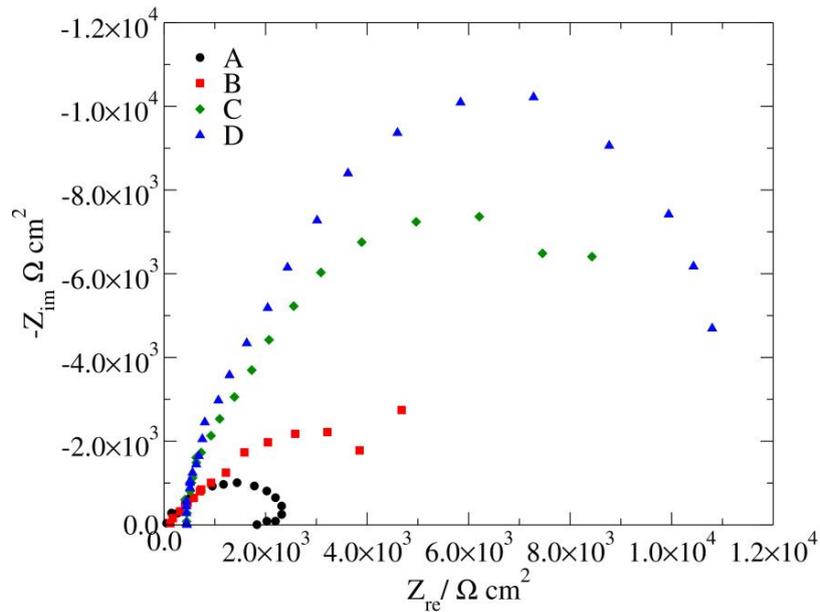


Fig. 5. The nyquist plots for as-deposited and heat treated (at 400 °C) Ni/P and composite coatings.

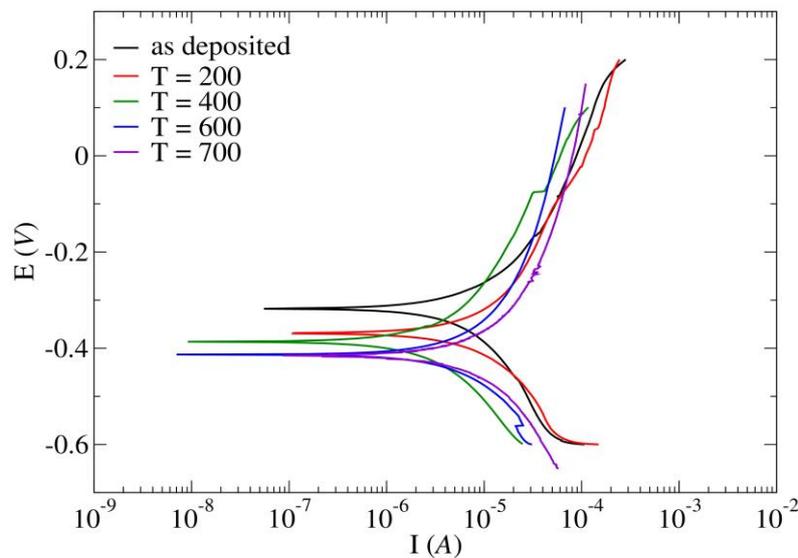


Fig. 6. Polarization curves for Ni/P coatings :(a) as deposited, (b), (c), (d) and (e) heated at 200, 400, 600 and 700 °C respectively.

but high temperatures caused Ni/P coatings to lose their resistance in corrosive environment. Heat treatment changes phase composition and causes phase transformation of Ni/P castings [45, 47] as XRD patterns show (Fig. 2), as-deposited coatings matrix presents a single broad peak which is related to an amorphous phase. After heating the coated samples, crystalline Ni and Ni₃P phases grow in the coating matrix and as

expected that corrosion resistance of the coating rises. Oxidation of Ni/P coating at higher annealing temperatures is responsible for lower corrosion resistance. Ni/P/TiO₂ nano-composite coating (as-deposited and heated at 400 °C) show better corrosion protection performance compared with plain coatings. The polarization curves for heated and as-deposited Ni/P/TiO₂ coatings are presented in Fig. 7.

The current density (i_{corr}) for as-deposited and heated composite coating was 2.4 and 0.61 $\mu\text{A}\cdot\text{cm}^{-2}$ respectively. When the TiO_2 ceramic particles are entered in the matrix of coatings, a passive layer is formed in the coating surface. Besides this, the TiO_2 particles are ceramic and non-reactive and have very high corrosion resistance and therefore the presence of TiO_2 particles in the matrix of Ni/P

coating increases the corrosion resistance.

Fig. 8 show the coefficient of friction of Ni/P, Ni-P-TiO₂ and Ni-P-TiO₂ heat treated at 400 °C. The results showed that the TiO_2 nano particles causes the surface of sample to be softer than other coatings. The wear resistance of the Ni-P-TiO₂ composite coatings had been increased with heating in 400 °C.

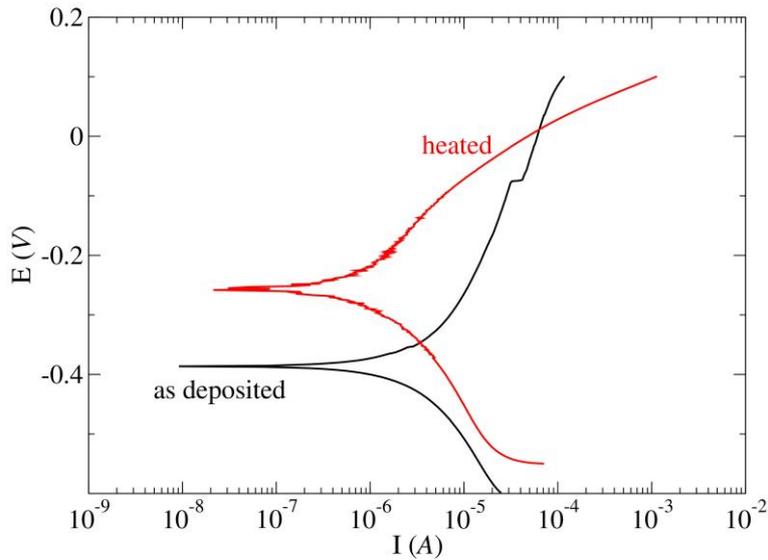


Fig. 7. Polarization curves for heated (at 400 °C) and as- deposited Ni/P/ TiO₂ coatings.

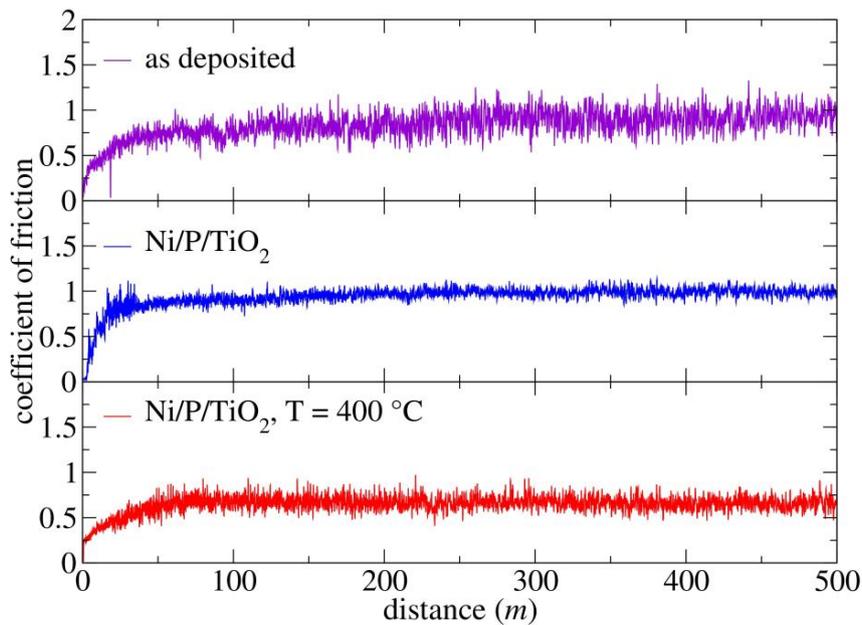


Fig. 8. Wear analysis for as deposited and heated Ni/P/TiO₂ (at 400 °C) and as deposited Ni/P coatings.

CONCLUSION

Ni/P and Ni/P/TiO₂ nano-composite coatings were obtained by electroless plating method. Inserting nano-particles into the matrix of Ni/P coatings improved the physical and mechanical performance of these coatings. In addition, the co-deposition of TiO₂ nano-particles caused an increase in the microhardness value of coatings and have changed the morphology of the deposit surface. Our results show that the maximum value of microhardness (1124 HV) and the highest wear and corrosion resistance were obtained at 400 °C for annealed composite coatings. The SEM and WDS data show a uniform and regular distributions of the nano-particles in the matrix of coatings.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this paper.

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