Effects of Addition of Waste Toner Particles on Tribological Properties of Ni-P Coatings Before and After Heat Treatment

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Abstract

This study investigated the tribological properties of Ni-P coatings produced by the electroless plating technique with various amounts of waste toner particles added to the plating bath. The properties were examined before and after a heat treatment process involving 60 minutes of heating at 400ºC. The impact of the concentration of toner particles in the plating bath on the properties and also the microstructure, phases, micro-hardness and wear mechanism and characteristics of the modified coatings were investigated. Field emission scanning electron microscopy (FESEM) images showed that toner particles were homogeneously distributed in the Ni-P matrix. The addition of 1 g/L of waste toner particles to the plating bath significantly increased the wear resistance of the coating and reduced its coefficient of friction (COF) to as low as 0.25. Although heat treatment increased the micro-hardness of all of the coatings, it did not lead to a significant improvement in the wear resistance of the toner-containing coating compared to the conventional coating. The presence of toner particles in the plating bath decreased the coating growth rate from 0.16 to 0.08 µm/min. Increasing the concentration of toner particles from 1 to 5 g/L decreased the toner content of the resulting Ni-P coatings, leading to reduced micro-hardness and consequently increased wear rate of the produced composite coatings.

Keywords: Ni-P Coatings; Toner; Electroless; Tribological Property; Wear.

1. Introduction

Electroless Ni-P coatings are commonly used in a variety of industries because of their excellent anti-corrosive property, catalytic characteristics, and mechanical performance [1)]. Over the years, researchers have developed various solutions for improving the properties of Ni coatings, including the addition of hard nanoparticles such as SiO_2 [2], SiC [3, 4], and Fe_2O_3 [1,5, 7)] as reinforcement agent and the addition of lubricant particles such as graphite[4)], graphene oxide[8)], MoS_2 [9, 12)], and carbon nanotube [13)]. However, these particles have limited practical use because of their cost. Thus, there is merit in finding cheaper reinforcement and lubrication agents for these coatings.

Toner is a substance used in laser printers and photocopy machines to form the printed text on the paper. This substance consists of carbon compounds (e.g. polypropylene and paraffin wax), amorphous silica, surfactants, release agents, iron dioxides, and other inorganic/organic additives [14,16)]. Once used, toner turns into waste toner particles, which can be potentially reused as a cheap reinforcement agent. Several studies have investigated the effect of reinforcement agents consisting of iron oxide particles

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and carbon compounds on the properties of Ni-P coatings. These studies are reviewed in the following.

In a study by Yang et al. [13], they produced Ni-P-single-walled carbon nanotube coatings by the electroless plating technique and investigated their properties. This study reported that the best tribological characteristics were achieved when the concentration of carbon nanotube was 1 g/L. Tamilarasan et al. [8] investigated effects of the addition of reduced graphene oxide (rGO) on the corrosion and erosion-corrosion properties of Ni-P coatings, ultimately reporting that the best properties for the Ni-P-rGO coating were achieved when the concentration of rGO particles in the bath was 0.05 g/L. In a study by Geng et al. [5], they investigated the effect of deposition parameters on the iron content in Ni-P-Fe_2O_3 electroless coatings. This study found that the best condition was achieved when the concentration of Fe_2O_3 particles was 16 g/L at a temperature of 65°C. Zuleta et al. [6] characterized the Ni-P-Fe_3O_4 coatings and studied their corrosion properties. This study found that, compared to Ni-P coatings, Ni-P-Fe_3O_4 coatings have higher corrosion resistance in 3.5 wt% NaCl solution at room temperature and a temperature of 800°C. In a study by Zhang et al. [1], they investigated the abrasion resistance and electromagnetic properties of electroless Ni-P-Fe_3O_4 coatings on polyethylene terephthalate (PET) fabrics. This study reported that the Ni-P-Fe_3O_4 coatings had lower abrasion resistance than the Ni-P coatings, a result that was attributed to the brittleness of the microcrystal structure of Ni-P-Fe_3O_4 coatings. A study by Shuang [7] suggested that the best corrosion resistance and the highest coating thickness could be achieved by setting the concentration of Fe_3O_4 nanoparticles in the plating bath to 0.8 g/L.

In this paper, as a new study, the toner powder as a cheap source that included both carbonic materials and iron oxides was used as reinforcement particles. Thus, the present investigation studied the effects of the addition of waste toner to Ni-P coatings on their tribological behavior, including micro-hardness, coefficient of friction (COF), and wear rate before and after the heat treatment process. The study also examined the microstructure of the composite coatings produced by adding different concentrations of toner particles to the plating bath.

2. Materials and Methods

2.1. Preparation of Composite Coatings

Ni-P composite coatings were deposited on St37 steel with the chemical composition C(0.18wt%), Si(0.33wt%), Mn(0.32wt%), P(0.05wt%) and S(0.05wt%). Before the deposition process, pre-treatment was performed as follows: grinding to 2500 grit, degreasing by acetone for 15 min in the ultrasonic bath, chemical degreasing in the NaOH solution for 4 min at 60°C, and acid pickling in 50% HCl solution. The Ni-P composite coatings were created by the electroless plating technique. Detailed information about the chemical composition of the Ni-P bath is provided in Table 1. The bath pH was maintained at 4.5-4.7 throughout the deposition process. The temperature range of the plating bath was about 83-87°C. The concentration of toner particles in the plating bath was set to 1, 2.5, and 5 g/L. For all of the coatings, the deposition time was about 90 min. As shown in Figure 1(a), the mean size of toner particles used in this study was 8 µm. As the X-ray diffraction (XRD) pattern illustrated in Figure 1(b) shows, these particles were consisting of carbon compounds, Fe_2O_3, and Fe_3O_4 particles. Toner particles were suspended in the plating bath by the sodium dodecyl sulfate (SDS) surfactant with a concentration of 0.5 g/L and then stirred by ultrasonic agitation at 600 rpm. A scheme of Ni-P-toner bath is observed in Figure 1(c). In the end, the heat treatment process was performed in a resistance furnace at a temperature of 400 °C for 60 min. This form of heat treatment is known to change the amorphous structure of the Ni-P coatings to a crystalline type [9, 17]. Detailed information about the heat-treated coatings is provided in Table 2.

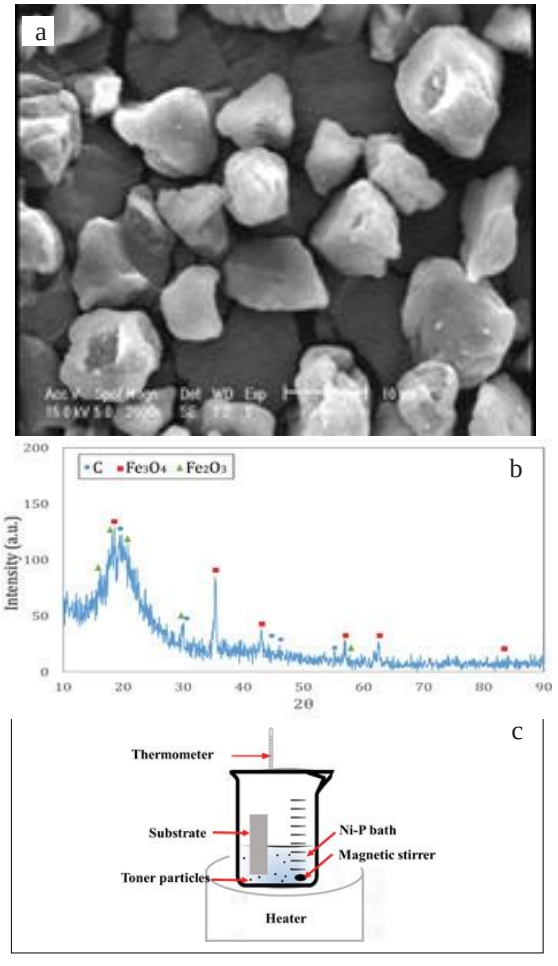


Fig. 1. (a) The SEM image, (b) the XRD pattern result of the used toner particles, and (c) a scheme of Ni-P-toner bath.

Table 1. Details of the chemical composition of Ni-P plating baths.

$NiSO_{4.6}H_2O$	$NaH_2PO_2.H_2O$	$C_6H_5Na_3O_7.2H_2O$	CH_3COONH_4
50 g/L	25 g/L	60 g/L	40 g/L

2.2. Characterization of Produced Coatings

The microstructure of the coatings was analyzed by a field emission scanning electron microscope (FESEM, MIRA3-TSCAN) equipped with an energy dispersive spectroscope (EDS). The phases in the coatings were investigated using an X-ray diffractometer (XRD, Bruker D8-Advanced) by applying Cu kα radiation over a scanning range of 10-90°. The micro-hardness of the coatings was measured by Vickers micro-hardness testing (Buehler) with a load of 0.01 kg. For each coating, this test was performed at least five times and the results were averaged to obtain the mean micro-hardness. Wear tests were performed using a pin-on-disk wear tester. In this test, the pin velocity was 0.12 m/s, the load was 15 N, the wear distance was 500m, and the humidity was 36%.. The pin used in this test was made of AISI 52100 steel with a hardness of approximately 800 HV. After the test, the worn surfaces were examined by a scanning electron microscope (SEM, Philips-XL30).

3. Results & Discussion

3.1. SEM Analysis and Thickness Measurement

The FESEM images illustrated in Figure 2 show the effect of toner particles on the surface homogeneity of Ni-P coatings before and after the heat treatment process. In Figure 2(a), the Ni-P coating is homogenous but has a number of nodules on the surface. The mean size of these nodules is lower than 3 μm. The presence of these nodules is evidence of the special growth mechanism of Ni-P coating as reported in [12]. In Figure 2(b), the coating with toner particles is clearly less homogenous than the previous coating. This difference demonstrates the effect of adding toner particles at a concentration of 1 g/L to the plating bath. The mean size of these nodules is over 5 μm. The growth of these large nodules on the surface must be attributed to the preferential adsorption of toner particles on the surface. Also, toner particles dispersed in the Ni-P matrix have a semi-cubical morphology (Figure 2(b)). Given the small size of nodules in Figure 2(c) and Figure 2(d), it seems that the surface roughness has decreased as the concentration of toner particles in the plating bath has increased from 1 to 5 g/L. The decline in the number of nodules on the coating surface could be due to a decrease in the toner content of the coating, as is also indicated in other works [8]. Overall, the Ni-P-5g/L toner coating exhibited the smoothest surface with fewest nodules (Figure 2(d)). With the increase in the concentration of toner particles in the plating bath, the matrix started to develop micro-porosity. This is because of the decrease in the coating density and the increase in its porosity, which result in hardness reduction [18]. As shown in Figures 2(e) to 2(g), heat treatment changed the microstructure of the coatings from amorphous to crystalline. It also turned the morphology of these coatings into a cauliflower-like structure. A similar effect is also reported in other studies [11, 14]. Also, as the toner concentration increased from 1 to 5 g/L, the grain size clearly increased and the grain morphology changed from semi-spherical to random. Other studies have also reported changes in morphology [19].

Table 2. Details all prepared coatings.

Coatings name	Toner concentration in plating baths (g/L)	Heat treatment process
Ni-P	0	No
Ni-P-1g/L Toner	1	No
Ni-P-2.5g/L Toner	2.5	No
Ni-P-5g/L Toner	5	No
Ni-P-H	0	Yes
Ni-P-1g/L Toner-H	1	Yes
Ni-P-2.5g/L Toner-H	2.5	Yes
Ni-P-5g/L Toner-H	5	Yes

Cross-sectional images of the coatings are illustrated in Figure 3. As can be seen, for a fixed plating temperature and pH, the addition of toner particles to the plating bath decreased the thickness of the produced coating.

The lowest and the highest thickness belonged to the Ni-P-5g/L toner coating and the Ni-P coating, respectively. As the concentration of toner particles in the plating bath increased to 5g/L, the solution viscosity also increased. This means a significant reduction in the movement of nickel ions toward the steel substrate, which leads to a decreased deposition rate. A similar observation is also made in another study [20]. The decrease in the deposition rate can also be attributed to the increased amount of surfactant present in the bath, which allows the surfactant molecules to cover the surface of the cathode, thus hindering the diffusion of nickel ions towards the surface and reducing their presence on the surface. This finding is also reported in other studies [17, 21]. Overall, as the concentration of toner particles increased, the thickness of the composite coating decreased from 13.1 to 7.5 µm. This means a 7-47% reduction in thickness (compared to the conventional Ni-P). Other studies have also reported a decrease in the deposition rate of Ni-P electroless coating in the presence of Fe_2O_3 particles at a concentration of 5-16 g/L [5]. For the deposition time of 90 min (which was fixed for all specimens), the Ni-P coating achieved the highest growth rate (~0.16 µm/min) and the Ni-P-5g/L toner coating showed the lowest growth rate (~0.08 µm/min).

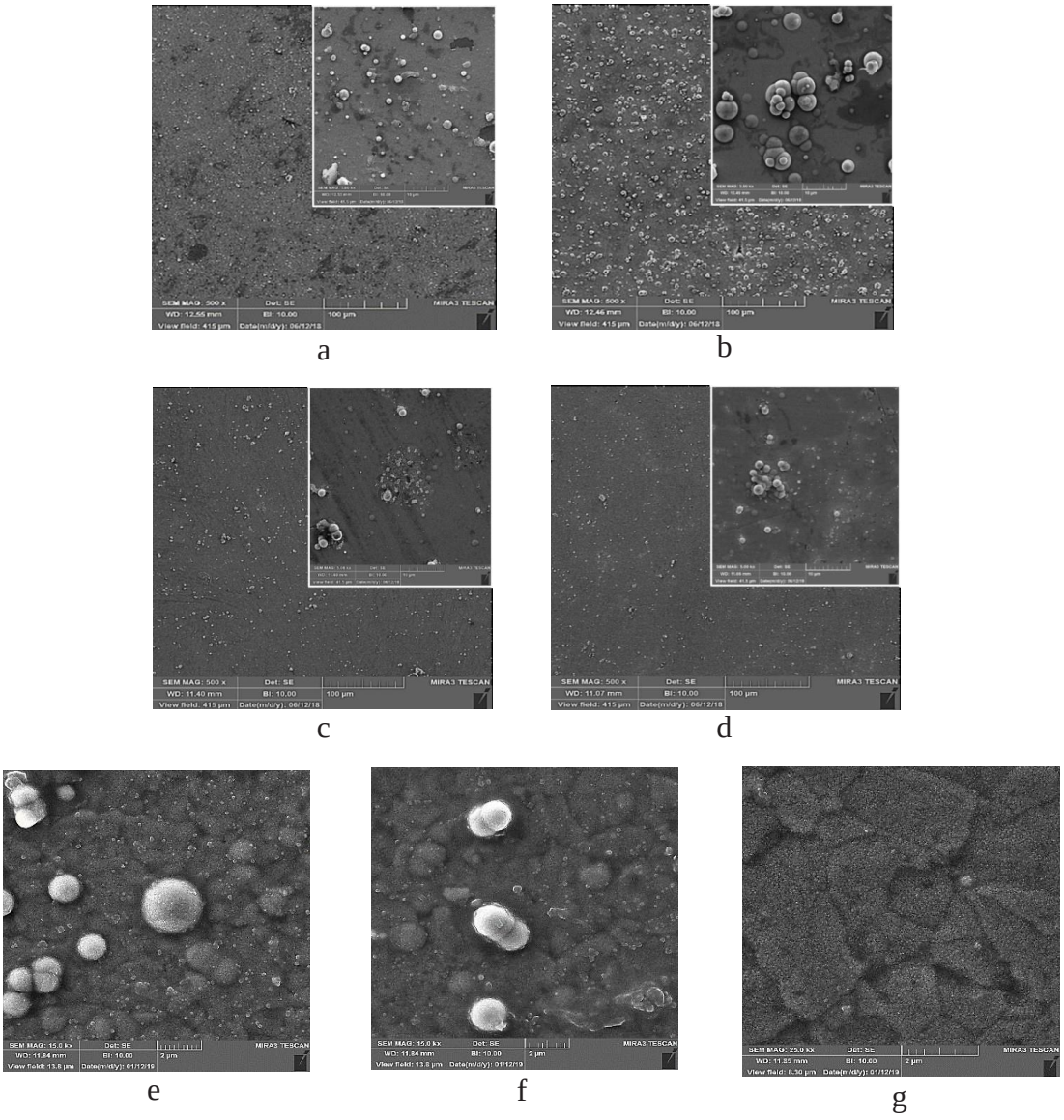


Fig. 2. FESEM images of various coatings from the top view, including (a) for the Ni-P coating in two magnifications, (b), for the Ni-P-1g/L Toner coating in two magnifications, (c) for Ni-P-2.5 g/L Toner coating in two magnifications, (d) for Ni-P-5 g/L Toner coating in two magnifications, (e) for the Ni-P-1g/L Toner-H coating, (f) for Ni-P-2.5 g/L-H Toner coating and (d) for Ni-P-5 g/L-H Toner coating.

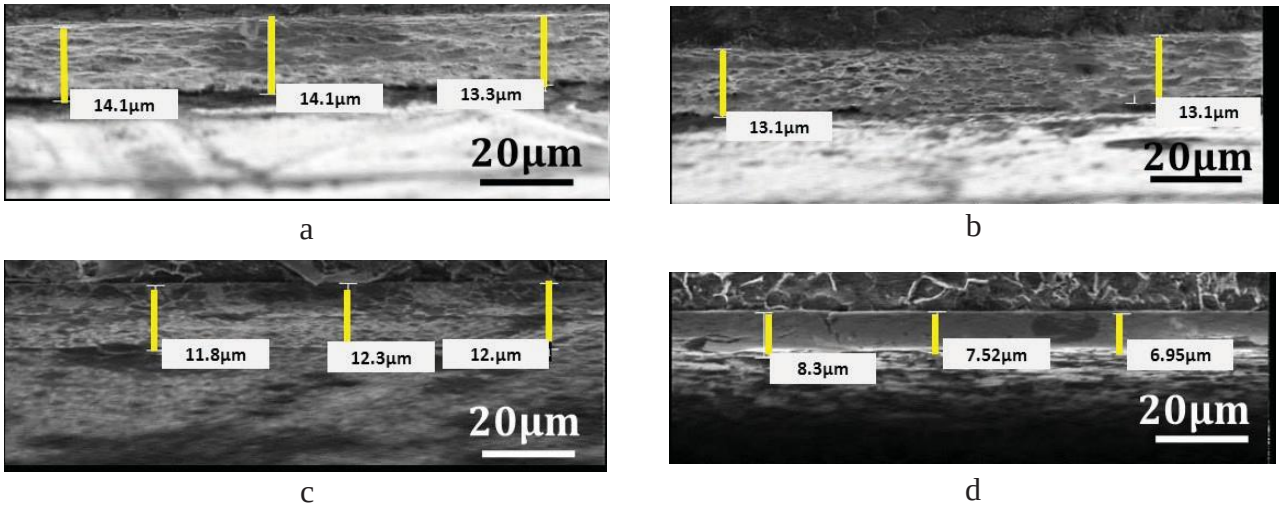


Fig. 3. FESEM images of various coatings from the side view, including (a) the Ni-P coating, (b) the Ni-P-1g/L Toner coating, (c) the Ni-P-2.5 g/L Toner coating and (d) the Ni-P-5 g/L Toner coating.

3.2. XRD and EDS Results

EDS and XRD analyses were performed to detect the phases present in the coatings. The results of the EDS analysis are shown in Figure 4. As can be seen, the addition of toner particles to the plating bath substantially decreased its nickel and phosphorus contents. It has been suggested that the presence of additional particles in the plating bath hinders nickel deposition by covering the surface areas [22]. The toner content of the coating was highest when the concentration of toner particles in the plating bath was 1 g/L. Also, as toner concentration in the plating bath increased to 2.5-5 g/L, toner particles got activated and started to agglomerate, which caused them to settle down, hence lowering the number of particles that can be fused into the Ni-P coating. Similar findings are also reported in other studies [13].

The XRD patterns of the coatings before and after the heat treatment process are displayed in Figure 5. Before the heat treatment, the Ni-P coating exhibited an amorphous phase (because of having broad peaks) consisting of Ni_5P_2 and Ni. The broadness of these phases was over the 2θ range of 35-55º. Zhang et al. [1] have also reported a similar observation in this regard. When toner particles were added to the plating bath, the pattern changed but not significantly. The peaks that appeared in this XRD pattern were related to Fe_3O_4 and Ni phases. As shown in Figure 5(a), the patterns did not show other peaks associated with toner particles (Fe_3O_4 and carbon compounds), probably because they were not present in amounts large enough to be detected. After increasing the concentration of toner particles to 5 g/L, the intensity of the Fe_3O_4 phase decreased. The presence of toner particles in the coatings also decreased the broadness of the peaks, limiting it to the range of 40-50º; a change that can be attributed to the decrease in the phosphorus content of the coating. Other studies have also reported similar findings [1] It was observed that the Ni-P coating with high phosphorus content exhibits an amorphous microstructure. Also, the broadness of the peaks decreased with the decrease of phosphorus content[1, 6, 2].

Figure 5(b) shows sharp peaks in XRD patterns, indicating that heat treatment has transformed the microstructure of these coatings into a crystalline phase. A similar finding has been reported in other studies [21]. The results suggest that heat treatment at 400 ºC for 60 min leads to the deposition of Ni_3P and Ni phases[18]. Because of the formation of the Ni_3P phase, all the coatings had high phosphorus contents. After adding toner particles to the Ni-P coating, the peaks related to carbon compounds and Fe_3O_4 also appeared in the XRD pattern. Considering the changes observed in the peak heights of various planes of the Ni_3P phase, the findings indicate a change in the preferred orientation of the coatings.

3.3. Micro-Hardness and Wear Properties

As shown in Figure 6(a), all the coatings that contained toner particles had a higher micro-hardness than the conventional Ni-P coating. This difference can be attributed to the dispersion strengthening effect of the particles [11, 12]. The hardness substantially decreased as the concentration of toner particles increased from 1 to 5 g/L. The results also suggest that having higher phosphorus contents in the coating results in lower hardness and higher wear [23]. Overall, the coatings with toner particles showed 1.3-3 times higher micro-hardness than the conventional Ni-P coating. The heat treatment process also augmented the micro-hardness of all the coatings. It has been reported that heat treatment leads to the precipitation of hard crystalline Ni_3P and Ni phases, which inhibit the dislocation mobility in the matrix, thereby increasing

hardness [1, 24]. The highest micro-hardness after the heat treatment was 1478 VHN and belonged to the Ni-P-1g/L toner coating. It was observed that the pH of the plating baths enhances the hardness of the produced coatings[25].

It was notable that although the porosity is increased insignificantly based on SEM images, the toner powder contained the iron oxide as ceramic particles, enhancing the hardness through the oxide particle strengthening.

Given the significant impact of the sliding distance and the applied force on the wear characteristics of the coatings [24], these parameters were kept constant in all wear tests. The specific wear rates of the coatings are shown in Figure 6(b). All of the coatings that contained toner particles showed lower wear rates than the conventional Ni-P coating. The decrease in the wear rate of these coatings ranged from 18 to 54% (compared to the conventional Ni-P coating). With the increase in the concentration of toner particles from 1 to 5 g/L, the wear resistance decreased. Consequently, the coating with the best wear resistance was the Ni-P-1 g/L toner coating.

This result should be attributed to the higher content of toner particles in the mentioned coating. The heat treatment process increased the wear rate of all coatings, an effect that can be related to the increase in the residual stress formation and brittleness and changes in the surface morphology of the coatings during the heat treatment process [26]. The carbon compound in toner particles decreased after heat treatment at 400ºC. Overall, the highest wear resistance after the heat treatment was related to Ni-P-1 g/L toner-H coating. In this regard, Ramalho et al.[27] reported that the said process cannot enhance the wear resistance of Ni-P-PTFE composite coatings.

Figure 7(a) and Figure 7(b) display the diagrams of COF versus wear distance of all the coatings before and after the heat treatment. Much like the wear rate results, before the heat treatment, Ni-P-1g/L toner coating had the lowest COF (0.25) among the coatings (Figure 7(a)). This can be attributed to the fact that this coating has higher toner content than others. Considering that toner particles contain carbon compounds and release agents, the presence of release agents near the surface of toner particles can reduce their coefficient of friction. Thus, the mean COF value for toner particles was lowered to 0.15 [28]. Thus, during the wear action, the shear force can disperse the toner particles and their release agent over the coating surface, resulting in reduced COF. Both before and after the heat treatment, the Ni-P coating had the highest COF among coatings. Also, the COF of this coating was higher before the heat treatment than after the treatment [21].

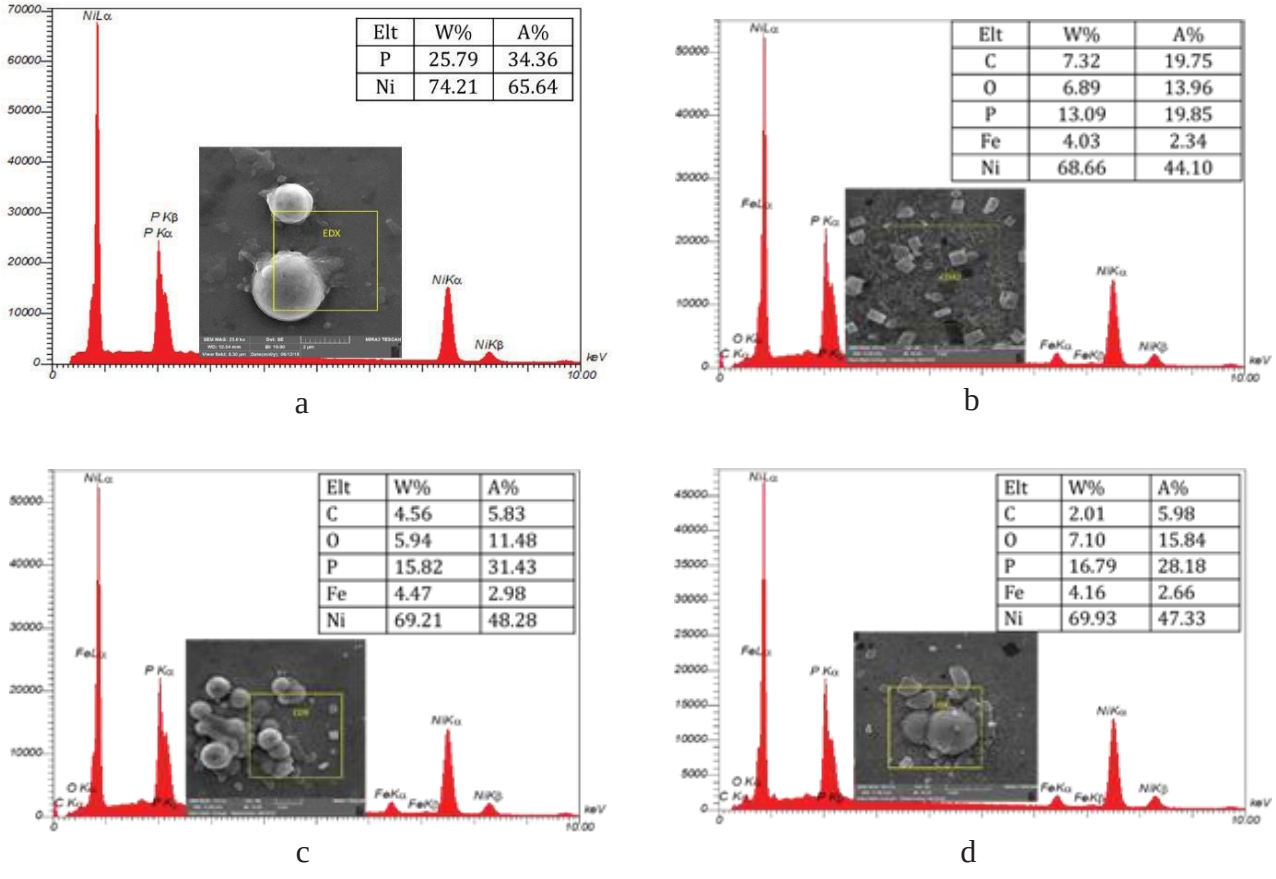
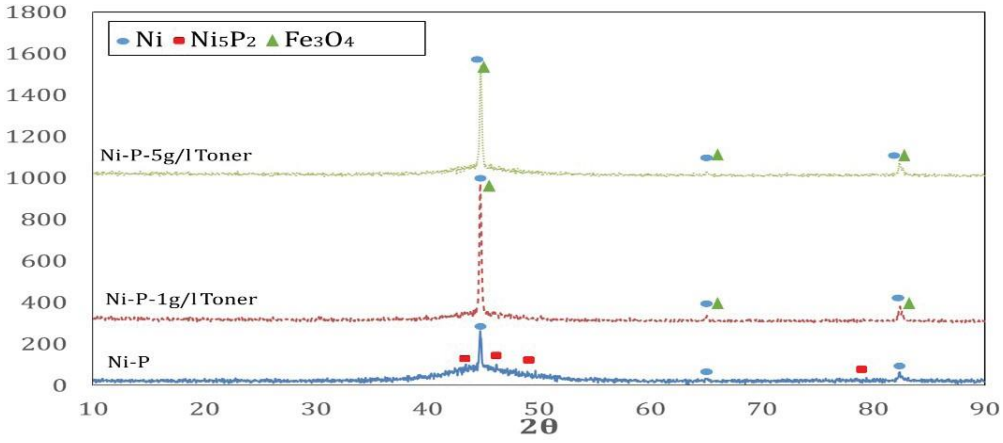
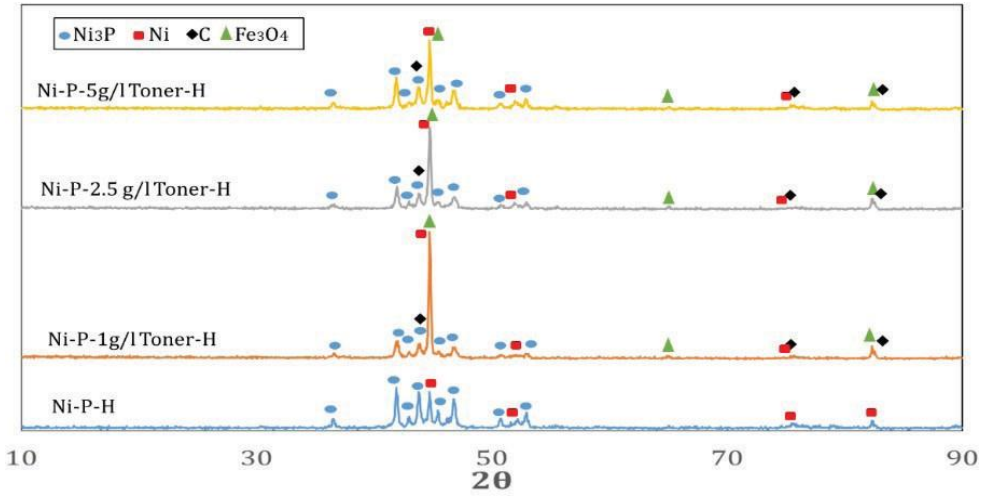


Fig . 4. EDS results for various coatings before the heat treatment procedure, including (a) the Ni-P coating, (b) the Ni-P-1g/L Toner coating, (c) the Ni-P-2.5 g/L Toner coating and (d) the Ni-P-5 g/L Toner coating.

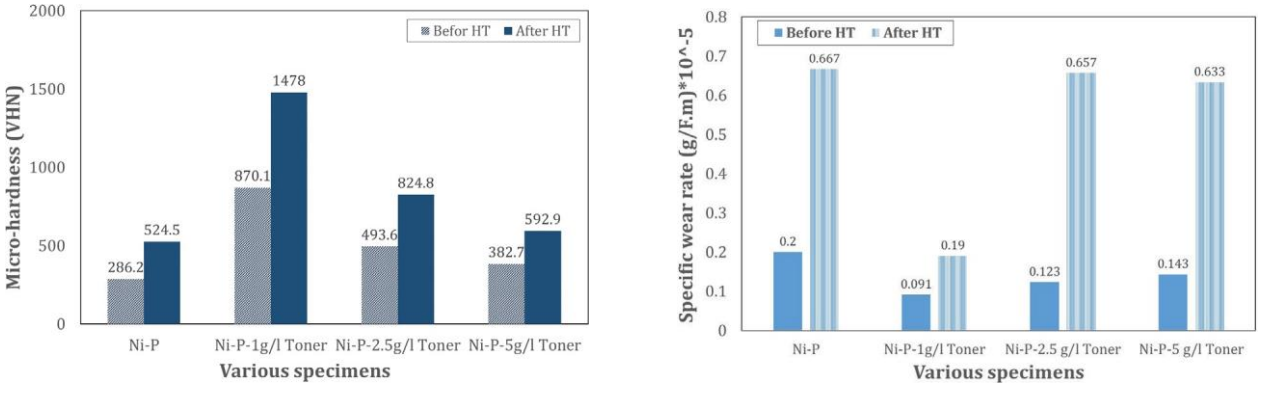


a



b

Fig. 5. XRD patterns for various coatings (a) before and (b) after the heat treatment process.



a b

Fig. 6. (a) The micro-hardness value and (b) the wear rate for various coatings before and after the heat treatment process.

As shown in Figure 7(b), the lowest COF belonged to the Ni-P-1 g/L toner coating after the heat treatment process, a result that is related to the higher hardness of this coating. When the concentration of toner particles was

raised above 1 g/L, COF increased so much that it became similar to that of the Ni-P coating. This is because of the reduction in the toner content of the coatings after the heat treatment process. The heat treatment decreased the COF of all of the coatings, but not significantly. The findings indicate that this can be attributed to the contribution of tribo-induced NiO formation after the heat treatment, which provides better solid lubrication capability during the wear test [26, 29]. The decrease in the size of surface nodules (Figure 2) also reduces the deviation of COF versus wear distance. Considering that the literature contains several approaches for predicting the wear rate of materials [23-32], the results were also examined from this perspective. This examination showed that the wear rates for most coatings can be predicted based on the ratio F/H (where F is the COF and H is the hardness of the coating).

SEM images taken from the coating surfaces after the wear test are shown in Figure 8. As Figure 8(a) shows, the Ni-P coating was homogenously flaked without any residual deep hole or cracking. This form of surface damage indicates a degree of plasticity. Therefore, the wear mechanism of the Ni-P coating appears to be of typical adhesive type. Other studies have also suggested a similar mechanism for this coating [19, 29, 33]. The wear width of this coating was approximately 1.8 mm. In the coatings containing toner particles, worn surface showed some micro-scratching and debris plus a number of pits and pores, which can be attributed to its higher hardness. Also, as shown in Figures 8(b) to 8(d), these coatings had lower wear widths than the Ni-P coating. The lowest wear width was 1.3 mm, which belonged to the Ni-P-1 g/L toner coating. The results also showed that the scratch depth decreased with the decrease in toner content. Hence, the toner-containing Ni-P coating appears to have an abrasive wear mechanism of the cutting type. The heat-treated coatings did not show homogeneous surface damage. The worn surfaces of these coatings showed signs of spalling, cracks, moderate scratches, and deep holes, which indicate higher wear rates. The heat-treated coatings also had 15 to 47% higher wear width than before the heat treatment. This change can be attributed to change in the microstructure of the coatings after heat treatment. The increased wear track width is another evidence showing the higher wear rate of the Ni-P coating [26]. Overall, the proposed wear mechanism for the produced coating is the abrasive wear of the micro-plowing mode. In general, increasing the concentration of toner particles in the Ni-P coating increased the rate of deep scratching on the surface, which resulted in an increased wear rate and COF.

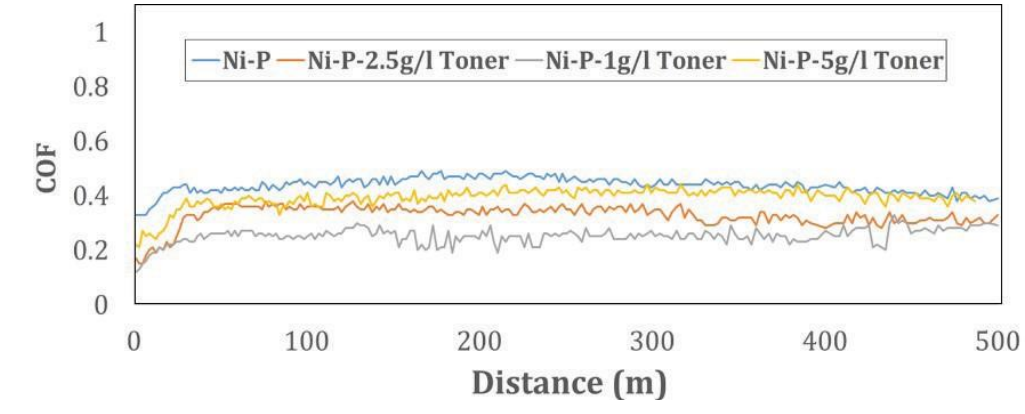


Fig. 7. The COF value versus the wear distance for all coatings (a) before the heat treatment process and (b) after the heat treatment process.

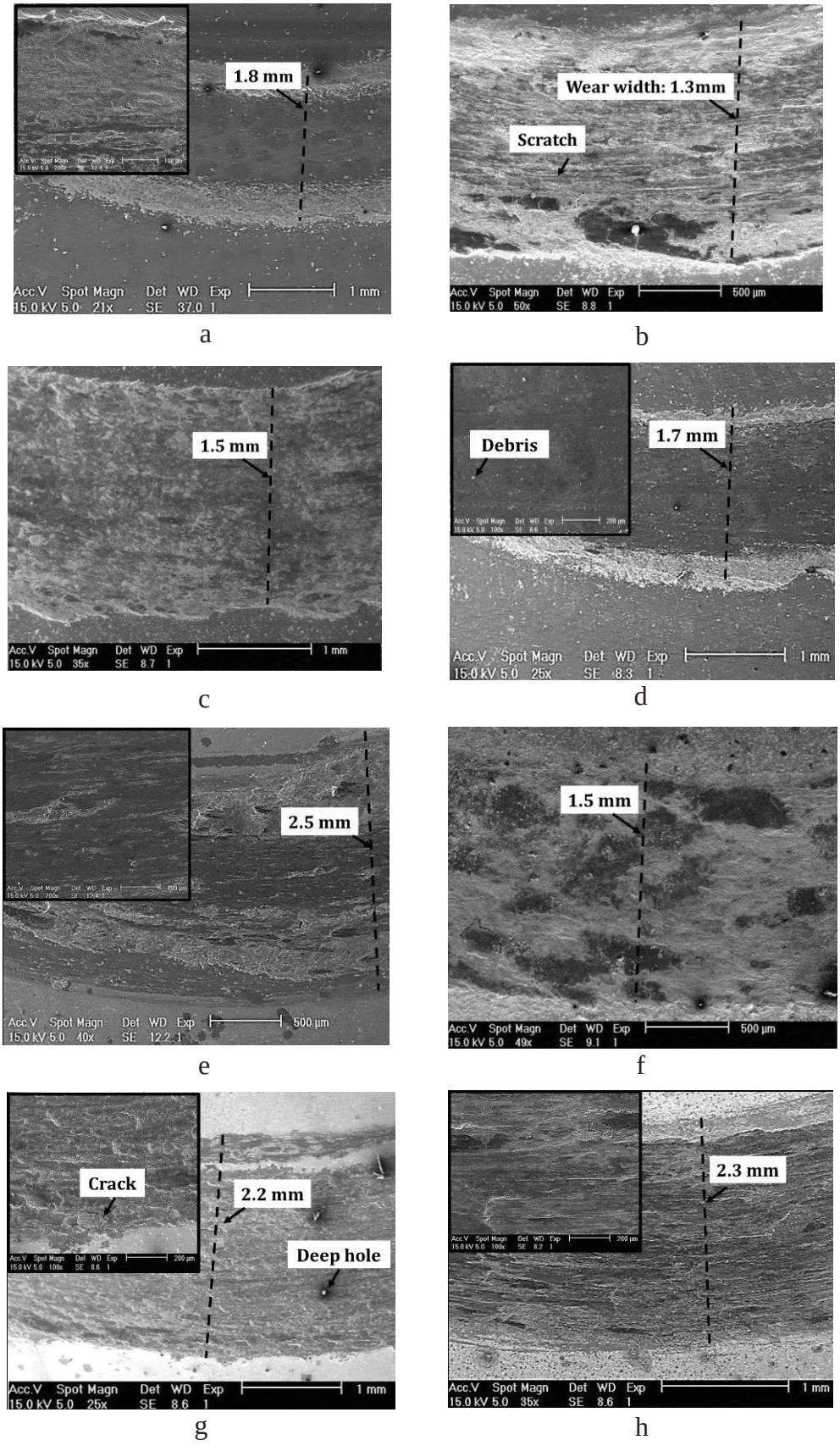


Fig. 8. SEM images of worn surface including (a) the Ni-P coating, (b) the Ni-P-1g/L Toner coating, (c) the Ni-P-2.5 g/L Toner coating and (d) the Ni-P-5 g/L Toner coating, (e) the Ni-P-H coating, (f) the Ni-P-1g/L Toner-H coating, (g) the Ni-P-2.5 g/L Toner-H coating and (h) the Ni-P-5 g/L Toner-H coating.

4. Conclusions

This study examined the tribological behavior of Ni-P coatings with waste toner particles. The main findings of the study are summarized below:

- The addition of toner particles to Ni-P coatings changed their characteristics. Scanning electron microscopy images demonstrated that toner particles were homogeneously distributed in the Ni-P matrix. When the concentration of these particles in the plating bath was increased to 5 g/L, the toner content of the Ni-P coating decreased.
- The presence of toner particles increased the micro-hardness of the Ni-P coatings up to 870 VHN. The heat treatment process further increased the micro-hardness of these coatings up to 1478 VHN.
- Before the heat treatment, Ni-P coatings with toner particles had significantly higher wear resistance than the coatings without these particles. The heat treatment led to an insignificant improvement in the wear resistance of these coatings relative to the conventional Ni-P coatings. Further, the presence of toner particles changed the wear mechanism from the adhesive mode to the micro-cutting abrasive mode.
- The addition of 1 g/L of toner particles to the plating bath decreased the COF of the produced Ni-P coating from 0.4 to 0.25. The lowest COF, 0.2, was observed in the heat-treated Ni-P-1g/L-H toner coating.

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