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ORIGINAL ARTICLE

Corrosion and wear properties of Ni-Sn-P ternary deposits on mild steel via electroless method

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Abstract The rising necessity to improve corrosion and wear resistance of metals for engineering applications cannot be over emphasized. This has led to employing diverse models, method and techniques to obtain better corrosion and wear resistances for metallic materials and components which will otherwise fail during service. This work investigated the effect of Ni-P binary and Ni-Sn-P ternary electroless depositions on the corrosion and wear behavior of mild steel. Microstructural examination using scanning electron microscopy (SEM) analysis shows finer and more evenly distributed particle orientation across the substrate surface. The Ni-Sn-P ternary deposits on the mild steel displayed from the linear polarization analysis a better corrosion resistance with corrosion rate values of 0.000246 mm/yr as compared with that of the Ni-P binary deposits with 0.016672 mm/yr. Also the coefficient of friction of the unplated sample varies between 0 and 0.08 while for the plated samples the coefficient of friction was relatively lesser and ranged from 0 to 0.02. Significant improvement in corrosion resistance was also indicated by a positive shift in potential. Sliding wear analysis demonstrates consistently enhanced wear resistance of the ternary deposits as well as the binary deposits, with the ternary Sn addition showing better resistance to wear. This work has established that Ni-Sn-P electroless coating of mild steel can be used to improve the corrosion and wear resistance for engineering applications.

1. Introduction

Surface engineering covers technologies proficient for modifying the surfaces of solids to afford advanced performance or innovative functionalities, with surface coating as a method applied widely in metal industries. Mild steel has wide usage in the manufacturing industry, however its vulnerability to corrosion and wear makes is imperative [1,2]. Corrosion effect is

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seen as the breakdown of materials reacting with an environment in which it is unstable [3,4]. Tribology is a mechanical phenomenon of critical concern in many types of machine components; in fact, it is often a major factor in defining or limiting the suitable lifetime of a component [5,6]. The consequence of corrosion and wear in the production, manufacturing and construction industries justifies the concerns accorded them.

Electroless plating has been implored for surface modification due to its immense benefits [7]. As a redox reaction, it depends basically on autocatalytic diminution of metal ions in aqueous solutions [8]. Electroless plating process is a common method used to produce homogeneous Ni [9] and its alloy depositions [10]. This process has been widely used to produce homogeneous, consistent coats for several industrialized applications. One major reason for the application of this technique is the high rate of deposition and it also affords the satisfactory result needed in production period at reasonable principal and functional overheads [11].

Several authors have reported a fine improvement of the corrosion rates of electroless nickel-phosphorous deposited metal [12]. Ni–P is a paradigm of electroless plating having extensive industrial uses as a result of the unique properties of its deposits. It has broad usage in machinery, electronics, and automobile, valve and aerospace industries because of its uniform coating and hence the attractive properties it evolves such as high hardness, good wear and corrosion resistances [13,14]. To broaden the applications of electroless Ni-P coating, a realistic and effectual technique to achieve superior electrochemical properties as an enhancement to that of binary Ni-P deposits is crucial [15].

The development of electroless quaternary or ternary alloy deposits is engaged. Various third addition models have been considered, and these consist but not limited to the following: Ni–Re–P, Ni–W–P, Ni–Cu–P and Ni–Zn–P [15–17]. Electroless and Corrosion of Nickel-Phosphorus-Tungsten alloy was studied by Ali Eltoum [18]. The Synthesis and Properties of Electrodeposited Ni-B-Zn ternary coatings were reported by Shakoor et al. [19]. The unique coatings obtained from the binary, ternary and quaternary electroless matrix have increased the drive to investigate possible alloy combinations that can be deposited on metals to manufacture beneficiated, efficient, effectual and improved metals [19]. Accordingly, many researchers have reported on tin as a third addition to electroless Ni-P plating examining its effect on different properties [20]. The effect of small additions of tin to the electroless Ni-P plating is studied, reporting improvement to the anti-corrosion competency in a 3.5% NaCl solution, yet a reduction of the same in sulfuric acid solution [21]. Ni-Sn-P electroless coat formation is examined, reporting that the ternary deposits showed enhanced anti-corrosion compared to the Ni–P deposits in shielding the plated sample in 10% HCl [22]. Tin addition to the electroless binary bath is investigated, focusing on the effect of free pre-activation technology, bath factors and operating conditions. The tin-nickel alloy gave a dense and less porous surface in contrast to the unevenly nodular surface of tin or nickel. In the open literature, conflicting reports have been given as to the significance of tin on electroless Ni-Sn-P plated metals with regard to corrosion resistance. This was attributed to the corrosive environment and coating composition. The need to contribute to knowledge on this corrosion property in other corrosive media could be essential in clearing conflicting reports. The product of tin addition on tribological wear properties of electroless plated mild steel has been meagrely examined. Other authors have investigated the effect of the addition of tin on other properties of the electroless deposits. These properties include the wettability, thermal stability, micro-hardness [17], crystallization and phase formation [23]. In this work, investigations will be carried out on the addition of tin to electroless Ni-P bath, with the aim of evaluating its effect on the corrosion resistance and tribological wear resistance of mild steel. Polarization analysis will be used to obtain corrosion results, and wear behavior of the electroless plated samples will be analyzed using a tribometer rotated under sliding pin with no lubricant at normal temperature.

2. Materials and methodology

2.1. Sample and pre-treatment

The material: mild steel substrate plate of dimension 0.5 m × 30 mm, with thickness 1.5 mm was obtained at the laboratory of Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa. The mild steel substrate was sectioned into smaller pieces of dimension 15 mm × 10 mm and drilled with a 2 mm drill so as to enable the sample to be held and immersed during the plating process. Pre-treatment of the samples was done by grinding with carbide paper until a mirror like surface is achieved. Samples were pickled in diluted H_2SO_4 acid solution; this helped to eliminate oxides and organic contaminants. The grinded and pickled samples were washed in distilled water after each stage.

2.2. Electroless bath formation

The electroless alloy depositions were performed by immersing the pre-treated mild steel sample in a glass beaker which contained 250 ml of the electroless plating solution. Table 1 lists the bath components and composition for Ni-P and Ni-Sn-P alloy depositions. All the bath components were obtained from Unified Scientific store Johannesburg South Africa. They were placed in a heating bath with the temperature monitored by a contact thermometer. The bath pH was adjusted with ammonium chloride acting as the buffer and agitation was set at 250 rpm.

Hypophosphite baths are mainly the broad type of commercially applied electroless nickel baths owing to improved stability, source of phosphorus in the deposit, higher plating

Table 1 Bath components and composition.

Component	Function	Amount (g/l)
Nickel chloride	Ni metal source	40
Tin (IV) oxide	Sn metal source	20
Sodium hypophosphite	Reducing agent	30
Trisodium citrate	Stabilizing agent	20
Ammonium chloride	Buffer	35
Ph		4–5
Temperature		80–90 °C
Plating time		30 min

rates and superior ease of bath control. Sodium hypophosphite was used as the reductant. Nickel (II) chloride, is one of the most important sources of nickel for chemical synthesis. Stabilizing agent regulates the rate of discharge of free metal ion for the redox reaction; trisodium citrate was used as the stabilizer and Ammonium chloride acted as the buffer.

2.3. Characterization

The microstructural analysis of the samples was prepared by cutting to rectangles of 15 mm × 15 mm and mounted with Bakelite resin. The samples were mechanically ground on 1000, 1200 grade SiC papers and polished using 3 μm cloths with diamond paste. Field emission scanning electron microscope (Model JEOL JSM-7600F) was used. The SEM was done in accordance with ASTM F137293(2012) Standard Test Method for Scanning Electron Microscope (SEM).

The X-ray diffraction (XRD) patterns of the samples were obtained with an X'PertPro PANalytical, LR 39487C. XRD diffractometer using Cu Kα radiation (40 kV, 40 mA). Stepwise increase for small angle was 0.01° over the range of 1–8° and wide angle rate of 1° $2\theta\,min^{-1}$ over the range of 8–90° (2θ).

For the corrosion analysis the sample was embedded in epoxy resin leaving a working area of 0.785 cm^2. The surface was ground with grinding papers from 800 to 1200 grit, cleaned with distilled water and ethanol. A conventional three electrode cell, consisting of saturated calomel electrode (SCE), graphite rod and coated mild steel was used as follows: reference, counter and working electrodes respectively. The electrochemical measurement was done with Autolab PGSTAT 101 Metrohm potentiostat/galvanostat. An electrolytic cell containing 50 ml of 3.65 wt.% NaCl solution, with plated sample, a graphite rod which works as counter electrode and of saturated calomel electrode as reference electrode was used. The potentiodynamic potential scan was fixed from −1.5 V to +1.5 mV with scan rate of 0.012 V/s. The electrochemical corrosion test was performed at room temperature in a static solution.

Wear tests were performed on the coated steel samples using the reciprocating CERT UMT-2 tribometer under dry reciprocating conditions with continual recording of the dynamic coefficient of friction values. A normal load of 25 N, sliding velocity of 2 m/s and 2 mm sliding distance were used. The samples of dimensions 1 cm × 2 cm were fixed securely in a fitted sample chuck. The coefficient of friction was recorded continuously during the test for duration of 1000 s of reciprocating movement.

3. Results and discussion

3.1. Substrate characterization

The chemical composition of the under studied substrate; mild steel is shown in Table 2. Fig. 1 shows the SEM/EDS spectra for the unplated substrate – mild steel. The EDS identified the elements present in the substrate to be Fe, Mn and C. The element with the major constituent and highest peak is Fe. The EDS analysis simply confirmed the chemical composition given in Table 1. From the SEM micrograph it can be seen that the substrate is a large-grains low carbon content. This is in par with the work of [18].

Table 2 Unplated mild steel's chemical composition.

Elements	Fe	Mn	C	Si	S	O
wt.%	98.40	0.05	0.10	0.18	0.04	0.05

3.2. Characterization of the plated mild steel

Characterization of the plated samples was also carried out using the SEM/EDS analysis. The SEM image and EDS of the binary and the ternary plated mild steel are shown in Figs. 2 and 3. The chemical reactions that must have occurred between the substrate material and the coating materials plus all compounds forming the bath must have produced new phases in the mild steel matrix. Coating that evolves good surface properties depends on the mechanism of homogenous structures, the absence of grain boundaries, no dislocation, compactness and denseness of the protective film formed.

The surface modification via the electroless Ni-P and Ni-Sn-P deposits on the substrate produced notable microstructural changes. The resultant uniformity and compactness in the coatings are commendable, indicating that it is free of the presence of defects such as stacking faults, disarticulation, and segregation and phase boundaries [24]. The morphology of the Ni-P deposits shows several large grains, circular in shape while that of Ni-Sn-P is a lot smaller in size but more like a strong rough wall. Addition of Sn to the Ni-P system displayed more compact, less porous and denser coating, as observed from the SEM micrograph in Fig. 3. The influence of Sn on Ni-P is visible in the morphology, grain refinement is evident, and this ultimately produces high strength in polycrystalline materials such as steel. It can be seen in Fig. 3 that the plated surfaces are filled with materials; the layers reveal significant inter-diffusion indicating good adhesion between the coatings and the surfaces of the substrate [17,23].

The elemental composition as revealed by EDS analysis can be seen from Table 3, and an assessment between the elemental composition of both plated samples, reveals that the weight percentages of the major constituents elements such as Fe, Ni, C and P were reduced making room for the Sn which took an approximate value of 10.12 wt.%. From physical metallurgy principles one can easily conclude that based on the SEM/EDS result the effect of tin should be positive on the properties of the coating since it resulted into grain refinement. This is in line with the previous work of [23].

XRD analysis showed the diffraction pattern of the Ni-P binary alloy has a major single Ni broad peak, which indicates that the structure of the plated Ni-P coating is amorphous nickel alloy. The result corresponds with the amorphous structure of nickel based coating also studied by [25–27]. A predominant Ni (1 1 1) peak and the presence of nickel phosphides Ni_3P are also observed as seen in Fig. 4a. Sn has remarkable effect on the characteristics of the Ni-Sn-P deposit as shown by the decreased intensity of the prominent peak. The addition of Sn to the binary Ni–P matrix produced crystalline phases [17,28,29]. This is probably due to the ultra fine grains of the Ni phase which further widened the peaks and increased the random dispersal intensity.

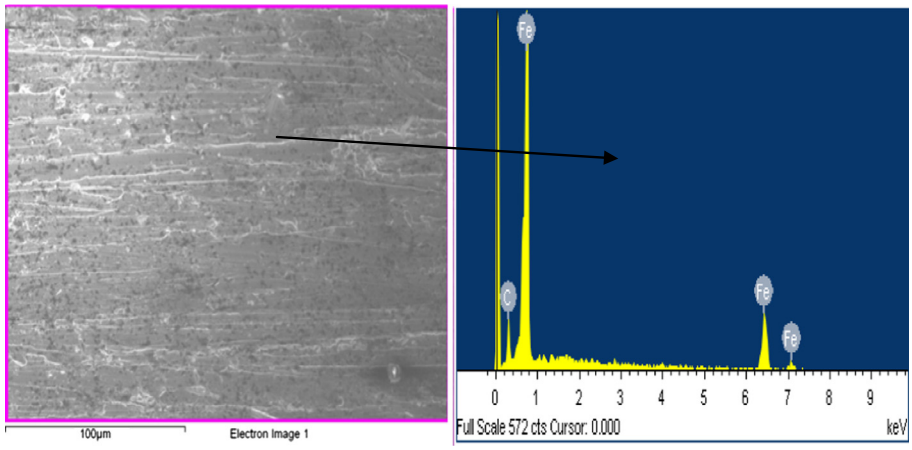

Figure 1 SEM/EDS spectra for the unplated mild steel.

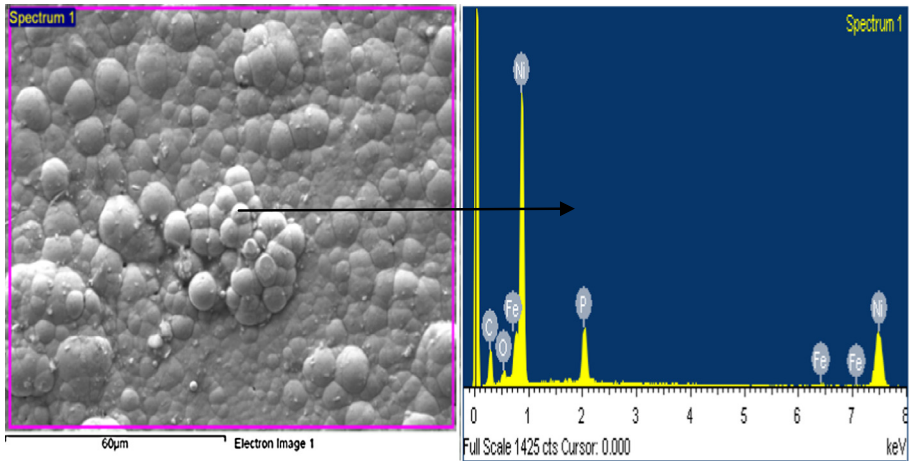

Figure 2 SEM/EDS micrographs of binary Ni-P coating.

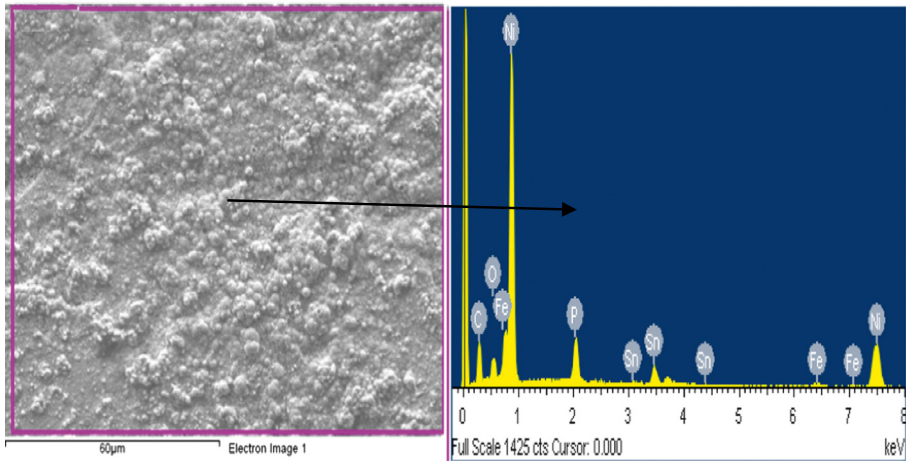

Figure 3 SEM/EDS micrographs of ternary Ni-Sn-P coating.

3.3. Evaluation of corrosion property of plated samples

The corrosion test data and the potentiodynamic polarization curves of all samples can be seen in Table 4 and Fig. 4 correspondingly. The unplated, Ni-P and Ni-Sn-P plated samples record corrosion potentials of −1.2151 V, −1.0515 V and −0.7296 V vs. SCE respectively. The corrosion potential E_{corr} of the plated samples shifted positively with 0.1636 V for the

Table 3 Elemental composition of the plated samples.

Elements	Ni-P (wt.%)	Ni-Sn-P (wt.%)
Fe	9.48	4.98
Ni	71.50	65.57
Sn	0.00	10.12
C	5.71	1.33
P	10.81	9.24
O	2.50	8.76

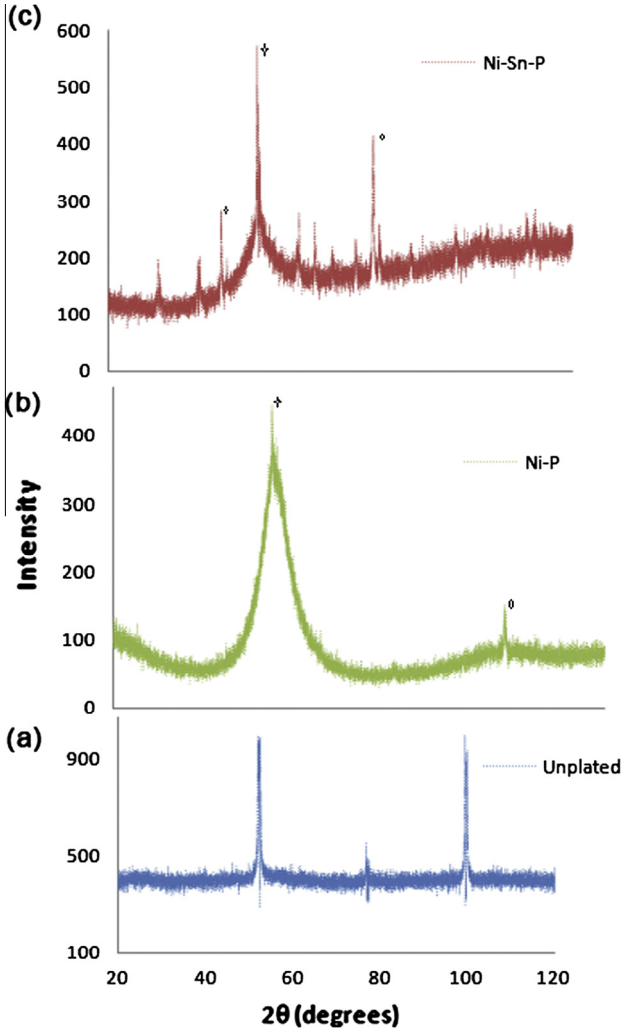

Figure 4a XRD Patterns for the (a) unplated, (b) Ni-P plated and (c) Ni-Sn-P plated.

Ni-P coat and 0.4855 V for Ni-Sn-P in contrast to the unplated. The increased E_{corr} values suggest that the plated samples are more corrosion resistant than the unplated sample.

The addition of Sn to the Ni-P coating however increased the E_{corr} by 0.3219 V. The polarization curves seem to indicate that the unplated and Ni-P plated samples in solution show evidence of slight passivation, and on the contrary Ni-Sn-P plated sample shows lack of passivation which is attributed to the presence of heterogeneous surface. The corrosion current densities (I_{corr}) of the unplated, Ni-P plated and Ni-Sn-P plated samples are 5.60E−05 A/cm^2, 2.42E−06 A/cm^2 and

Table 4 Electrochemical polarization data.

Sample	E_{corr} (V)/ SCE	I_{corr} (A/ cm^2)	Corrosion rate (mm/yr)	Polarization resistance (Ω)
Un-plated	−1.2151	5.60E−05	0.385580	703.53
Ni-P plated	−1.0515	2.42E−06	0.016672	1440.89
Ni-Sn-P plated	−0.7296	2.41E−08	0.000246	2948.09

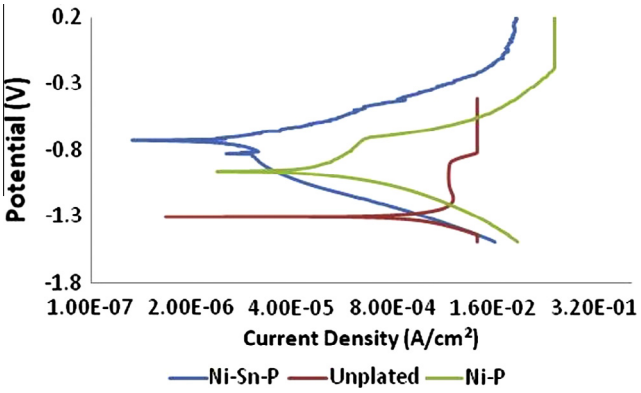

Figure 4b Linear polarization curve for unplated, Ni-P and Ni-Sn-P plated mild steel.

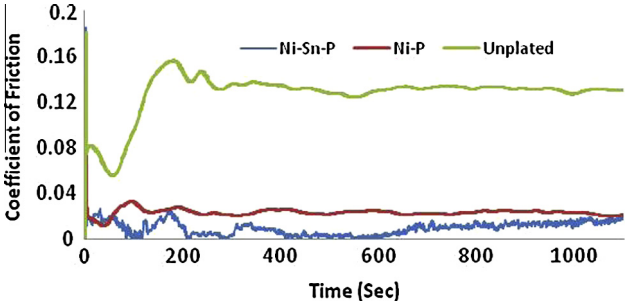

Figure 5 Plot of coefficient of friction vs. time at ambient temperature.

2.41E−08 A/cm^2 respectively. The coatings were much more corrosion resistant than the substrate because the anodic current density is much lower for the unplated mild steel. A three order and a two order magnitude reduction in (I_{corr}) was achieved subsequent to the binary and ternary electroless plating. The plated samples displayed corrosion potentials which were found to move toward the positive side as is visible from Fig. 4b.

The improvement observed was credited to the oxide film formed on the plated samples. From EDS result, oxygen was found to be present in the ternary Ni-Sn-P coating which is approximately 8.76 wt.% and this is a sign of the formation of oxidized films on the plated surface. NiO and SnO oxide layers are chemically stable phases and are valuable obstacles on the plated surface against corrosion attacks from the $C1^-$ containing aggressive medium. From polarization data, a cor-

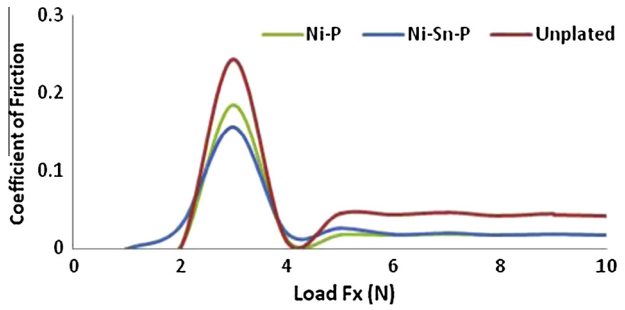

Figure 6 Plot of coefficient of friction with load at ambient temperature.

rosion rate of 0.000246 mm/yr for the Ni-Sn-P ternary alloy deposition was reached as against a corrosion rate of 0.016672 mm/yr from the binary alloy deposits. A two order decrease in magnitude occurs in corrosion rate as a result of Sn addition. The reduction in corrosion rate positively indicates an enhancement to the anti-corrosion ability of the mild steel. Considering all the corrosion parameters evaluated - R_p, E_{corr}, I_{corr} and the corrosion rate, Ni-Sn-P exhibits highest corrosion resistance in the test solution. Conclusions can be made that from the polarization analysis, Ni-P deposition shows improved corrosion resistance and a better corrosion resistance value is observed from the ternary Ni-Sn-P alloy deposition indicating more resistant of the material to corrosion sequel to the addition of Sn.

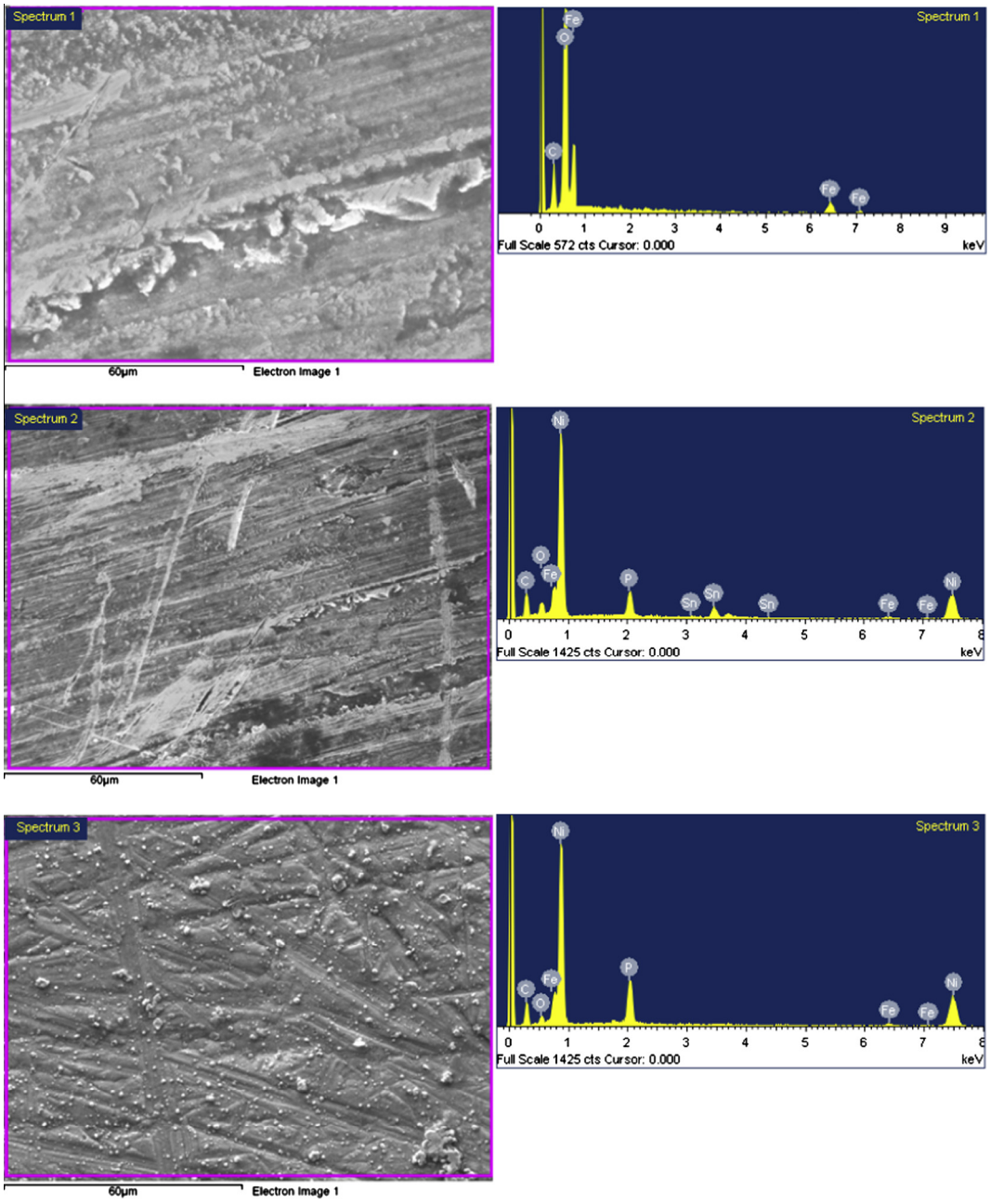

Figure 7 SEM micrographs for the worn surfaces: spectrum 1 unplated, substrate 2 Ni-P plated and 3 Ni-Sn-P plated.

3.4. Evaluation of wear resistance analysis

Fig. 5 shows the coefficient of friction for the unplated and coated Ni-P and Ni-Sn-P with respect to time. The coefficient of friction for the uncoated mild steel at ambient temperature varies between 0.08 and 0.16. For the electroless plated Ni-P and Ni-P-Sn samples conversely, the coefficient was found to be relatively lesser, ranging from 0 to 0.023. This optimum lower range can be accredited to the presence of oxide formations such as SnO, NiO, PO_2 and phosphides such as Ni_3P. In addition, the improvement in the wear depth analysis of the electroless plated sample as to the unplated can be attributed to greater affinity that exist between Sn, Ni, P and oxygen. Thus this enhanced the oxide layer as well as improved the bonding between the substrate and coating.

The coefficient of friction for the unplated mild steel and plated samples at different loads is shown in Fig. 6 below. For the unplated sample, the coefficient of friction varies between 0 and 0.08 while for the plated samples the coefficient of friction was relatively lesser and ranged from 0 to 0.02. The coefficient of friction got to its peak values at regions between 2 N and 4 N. The peak values for the samples are 0.245 for the unplated mild steel; 0.185 for the Ni-P plated sample and 0.157 for the Ni-Sn-P plated sample, indicating that the ternary deposition had better resistance to wear as the frictional load varied. The decrease in coefficient of friction is due to the increase in oxides on the Ni-Sn-P plated sample, thus signifying the protection which the electroless coatings render to the mild steel plate.

Fig. 7 shows the SEM micrographs for the scratched surfaces of the substrate, Ni-P and Ni-Sn-P plated mild steel. The evidence of wear rubble pits and slight groove which points to plastic deformation, visible on its worn surface with a lot pulling off can be seen from the worn surface of the sample material. On the contrary, mild wear is observed for the Ni-P and Ni-Sn-P plated samples, attributed to the good adhesion produced by the electroless coatings.

Micrographs of the worn surfaces indicate no split, fine scratches and little scales for the Ni–Sn-P coatings which can be seen to have undergone abrasive wear. However, some miniature bulges could be seen as the abrasion channel terminated. It can be drawn from the results of the graphs, figures and surface morphology that ternary Ni-Sn-P alloy deposition improved the tribological wear resistance of the metal, sequel to the infusion of Sn to the binary electroless bath.

4. Conclusions

From the results and discussion above the following conclusions can be made:

1. The unplated, Ni-P and Ni-Sn-P plated samples record corrosion potentials of −1.2151 V, −1.0515 V and −0.7296 V vs. SCE respectively.
2. The sliding wear analysis shows considerable increase in the wear resistance of the mild steel substrate, attributed to the electroless deposition.
3. The microstructure developed after Sn addition was the major reason for the improvement of corrosion and wear resistance of the mild steel.
4. The integration of metals with good corrosion and wear properties on the electroless bath produces deposits with striking qualities consequently improving the electrochemical and physical properties of the mild steel. This consequently fosters manufacturing processes involving the application of mild steel.

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