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RESEARCH ARTICLE - MECHANICAL ENGINEERING

Estimation of Zwitterionic Surfactant Response in Electroless Composite Coating and Properties of Ni–P–CuO (Nano)Coating

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Abstract
Electroless Ni–P and Ni–P–CuO coating on mild steel was developed successfully with the addition of Zwitterionic surfactant. The usage of nano-CuO in electroless coating was intermittent though the cost is low with high catalytic activity. Zwitterionic surfactant was introduced into the composite coating for the first time to increase the suspension of nanoparticles effectively during the coating process. Surfactant helps to reduce the intermolecular attraction between the solid and liquid interfaces and hence binding of nanoparticles with the hydrogen gas bubbles was eliminated. Also agglomeration of nanoparticles was controlled by stirring the electrolyte continuously using magnetic stirrer. The characterization and tribological properties were tested for the newly developed composites. Scanning electron microscope micrograph reveals the deposits are produced without any defects and the presence of CuO nanoparticles in the deposit. Energy-dispersive spectroscopy measurement shows the changes of weight percentage of elements available in the substrate. The surface roughness of the deposit was improved with the addition of CuO, it packs the gap between two grains and offers smooth finish and as the result the surface properties are modified. Microhardness of the substrate was improved for the substrate added with nano-CuO. The corrosion resistance of the substrate was improved when compared to the substrate produced using electroless Ni–P binary coating. Zwitterionic surfactant reduces the agglomeration of nanoparticles during chemical reaction and allows the particles to coat only on the target. Similarly, this technique can be implemented in the production of other composites in electroless coatings.

Keywords Composite coating · Zwitterionic · Roughness · Microhardness · Corrosion

1 Introduction

Mild steel is used commonly in industrial applications due to its good mechanical properties, machinability, availability and low cost. Corrosion is the critical problem faced by engineers, which may be chemical or electrochemical that relies upon the nature of the environment used. The huge cost is spent to replace the degraded materials for maintaining the physical and mechanical properties of the material [1]. The common way to protect the metals from corrosion is covering the surface using high corrosion resistance or composite materials. Variety of methods were found to deposit the protective layer such as paint coating, galvanization, chemical vapour deposition, physical vapour deposition, chrome plating, electroplating and electroless plating. Among these, electroless plating is utilized in numerous commercial enterprises with novel attributes such as uniform deposits, high resistance to wear and corrosion. The binary, trinary and composite coating can be prepared effortlessly without any major modifications in the experimental setup. Electroless plating is a redox reaction process, which deposits metal particles on the substrates without the aid of electric current [2,3]. The quality of the deposit relies on the amount of phosphorous substance present in the deposit [4]. Low phosphorous (2–5 wt.%) in the deposit improves hardness and wear resistance

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whereas above 10 wt.% in the deposit, referred to as high phosphorous, improves the corrosion resistance of the substrate [5]. To boost the properties such as hardness, wear, and corrosion resistance, a metallic or non-metallic element is co-deposited with the Ni–P binary alloy coating.

Earlier researchers reported that the incorporation of nanoparticle such as SiO_2 [6], TiC [7], carbon nanotubes (CNT) [8], Ag [9], SiC [10] Al_2O_3 [11] and TiO_2 [10] particles into electroless Ni–P process improved the corrosion and wear resistance of the substrate by covering the surface. The co-deposition of copper into Ni–P matrix increases the corrosion resistance of substrate [12]. This co-deposition of nanoparticle enhances the properties of the composite coatings particularly in wear and corrosion properties. Surface active agent referred to as surfactant improves the stability of particle suspension in the electrolyte during chemical reaction by increasing the wettability [13]. Sahar Afroukhteh et al. [7] studied the impact of various sorts of surfactant addition in the Ni–P–TiC bath and reported that corrosion resistance was improved. Surfactant also builds co-deposition of particle which is added in the electrolyte especially in the presence of anionic surfactant. The deposition rate of the process is moderately low after incorporating the surfactant into the bath. Zarebidaki et al. [14] examined the effect of surfactants to disperse the CNT particles added in the electrolyte during the coating process. Results showed that sodium dodecyl sulphate (SDS) dispersed consistently all through the deposit, which improves the corrosion resistance by filling the pores and micro-holes in the Ni–P coating. The deposition rate was improved significantly by introducing Zwitterionic surfactant in binary Ni–P coating process and the critical micelle concentration (CMC) value of 0.018 g/L was reported. Also decomposition of electrolyte never appeared even after 4 h of deposition process [15]. Ansari and Thakur [16] studied the effect of cationic surfactant in composite coating and improved the corrosion resistance of the magnesium substrate. From the results, it was concluded that the cationic surfactant has the strong influence on improving the corrosion resistance and nanoparticle distribution. From the extensive literature survey, it is observed that there is no relevant research data reported on the electroless coating of Ni–P–CuO deposits on low-carbon steel substrate with the addition of Zwitterionic surfactant, which is worthy of importance in this research. So far, the effect of Zwitterionic surfactant on the dispersion of nanoparticles in the composite coating was not reported.

This research is mainly focused on the development and the effect of adding nano-CuO particles and Zwitterionic surfactant in the electroless coating process. Earlier researchers have not used the nano-copper oxide particles as reinforcement. Zwitterionic surfactant which has significant effect on improving the deposition rate, dispersion and tribological properties were also not studied. The newly formulated deposits are analysed for their morphology, structure, surface roughness, microhardness and corrosion resistance.

2 Experimental Details and Procedure

The mild steel (0.14 wt.% carbon, 0.35 wt.% manganese, 0.17 wt.% silicon, 0.025 wt.% sulphur and the remaining iron) samples were prepared cylindrically having a size of 20 mm diameter and 7 mm thickness. The samples were mechanically polished by surface grinder followed by disc polishing machine. Pre-treatment was done on the substrate to evacuate the impurities and to activate the substrate surface before the start of the coating process. Three essential steps were followed in pre-treatment process as (i) de-greased using acetone for 3 min, (ii) cleaning with ethanol for 3 min and (iii) dipped in 20% H_2SO_4 solution for 30 s. In every stage, samples were rinsed using deionized water. Finally, it was placed into the prepared electrolyte for the coating process. The compositions and operating conditions for the electroless coatings are given in Table 1.

Analytical grade nickel sulphate, sodium hypophosphite, sodium citrate, ammonium acetate were used as a source of nickel, reducing agent, complexing agent and buffering agent, respectively. Zwitterionic surfactants, namely 3-(N,N-di-methyl myristyl ammonia) propane sulphonate (C14-SB) and copper oxide nanoparticle (< 50 nm particle size) were procured from Sigma-Aldrich and used in the coatings. pH of the electrolyte was maintained at 8 using liquid ammonia solution. The electroless coatings were carried out in a 250-mL glass beaker at 85 °C for 1 h. The Zwitterionic surfactant was weighed to 0.018 g and mixed with 1 L of distilled water. Then magnetic stirrer was used to mix it thoroughly. After the start of reaction in the electrolyte, 5 drops of prepared surfactant were added using pipette. During co-deposition of nano-copper oxide, the electrolyte was stirred magnetically to maintain the suspension of nanoparticle throughout the process. The simplified form of the reaction during the coating process is shown in the following equations:

Anodic reaction

$$H_2PO_2^- H_2O \rightarrow H_2PO_3 + 2H^+ + 2e^-, E^0 = 0.50\,V \quad (1)$$

Cathodic reaction

$$Ni^{2+} + 2e \rightarrow Ni^0, E^0 = -0.25\,V \quad (2)$$

$$2H^+ 2e \rightarrow H_2, E^0 = 0.000\,V \quad (3)$$

$$H_2PO_2^- 2H^+ e \rightarrow P + 2H_2O, E^0 = 0.50\,V \quad (4)$$

After coating, the specimens were rinsed with deionized water for 5 s, dried and preserved for further testing. The procedure to estimate the deposition rate of coated substrates was followed according to the DIN 50988 standards [13]. The surface morphology of the deposits were observed

Table 1 Chemical composition and operating conditions of electroless bath

Chemicals		Composition (g/L)		
		Ni–P	Ni–P–CuO	Ni–P–CuO (without surfactant)
$NiSO_4 \cdot 6H_2O$	Nickel (II) sulphate	30	30	30
$NaH_2PO_2 \cdot H_2O$	Sodium hypophosphite	40	40	40
$(NH_4)_2SO_4$	Ammonium sulphate	50	50	50
$C_6H_9Na_3O_9$	Sodium citrate	25	25	25
C14-SB	Zwitterionic surfactant	0.018	0.018	–
CuO	Copper (II) oxide	–	1	1

using scanning electron microscope (SEM) with a voltage of 15 kV (Hitachi, Model: S-3400N), and an energy-dispersive spectroscopy (EDS) attachment was used for element identification in the deposit. The structure of deposits were characterized using X-ray diffraction (XRD), Rigaku Ultima IV X-ray diffractometer with Cu anode. The step scanning rate was 0.02/s ranging from 10° to 90°. Retractable type surface roughness measurement tester (model: SJ-210) was used for analysis. The evolution profile was recorded in the computer using Surftest SJ USB Communication Tool software (Version 5.006). ASTM E92 standard was followed to measure the microhardness in the deposit. Applied load was 200 g and five readings were taken at random location and the average was reported as microhardness value. According to the ASTM G31 standard, immersion corrosion test was conducted on the developed samples. The coated substrates were rinsed with deionised water, cleaned with acetone and deionised water consecutively. Initial weight of the substrates was measured using weighing machine with the accuracy of 1 mg. Then, the substrates were dried and immersed in 3.5 wt.% of NaCl solution at room temperature for 25 days. The substrate was cleaned, drained and the weight loss was determined at the end of every 5-day interval (5, 10, 15, 20, and 25 days).

3 Results and Discussion

3.1 Deposition Rate and Working of Zwitterionic Surfactant

The deposition rate of Ni–P matrix is 34.38 μm/h as shown in Fig. 1.

It is reported from the present study that the addition of nano-copper oxide particles reduces the deposition rate. This could be attributed when the substrate (cathode) surface was covered by CuO particles which reduces the distribution of the nickel ions towards the active region and as a result, the deposition rate was affected [7]. The deposition rate of the composite coating without surfactant was measured to be

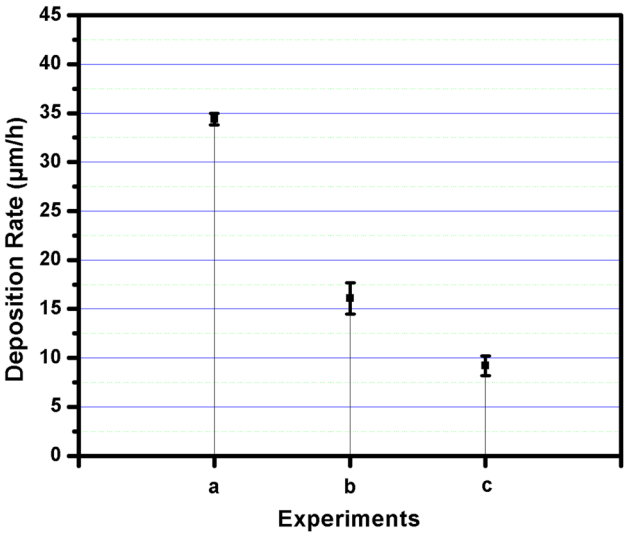

Fig. 1 Deposition rate obtained from electroless coating. **a** Ni–P **b** Ni–P–CuO **c** Ni–P–CuO without Zwitterionic surfactant

9±2 μm/h for the same chemical composition as represented in Table 1. After the addition of Zwitterionic surfactant, it increased the deposition rate to 20 ± 4 μm/h. A monomer layer was formed by added Zwitterionic surfactant, which comprised of the positive and negative head. Negative head of the surfactant monomer attracted more nano-metallic particles and pulled all the nanoparticles towards the substrate. Nanoparticles were also facilitated with repulsive force from the positive head of surfactant, which restricted the particle to cross the monomer layer after it reached the boundary. The deposition rate for composite coating was very low without Zwitterionic surfactant [15,17]. The surfactant allowed the nickel particle to coat only on the substrate surface which enhanced the deposition rate on the substrate. Also it reduced the surface tension between the metallic particles and hydrogen bubbles which was evolved during the coating process. The adsorption of tiny H_2 bubbles at the surface of a plated Ni–P deposit was regarded as the main cause of pinholes or pits [18]. Hence, the major drawback of pinholes, pits and binding of metallic particles to the hydrogen bubbles were eliminated. Due to very low surface tension in the pre-

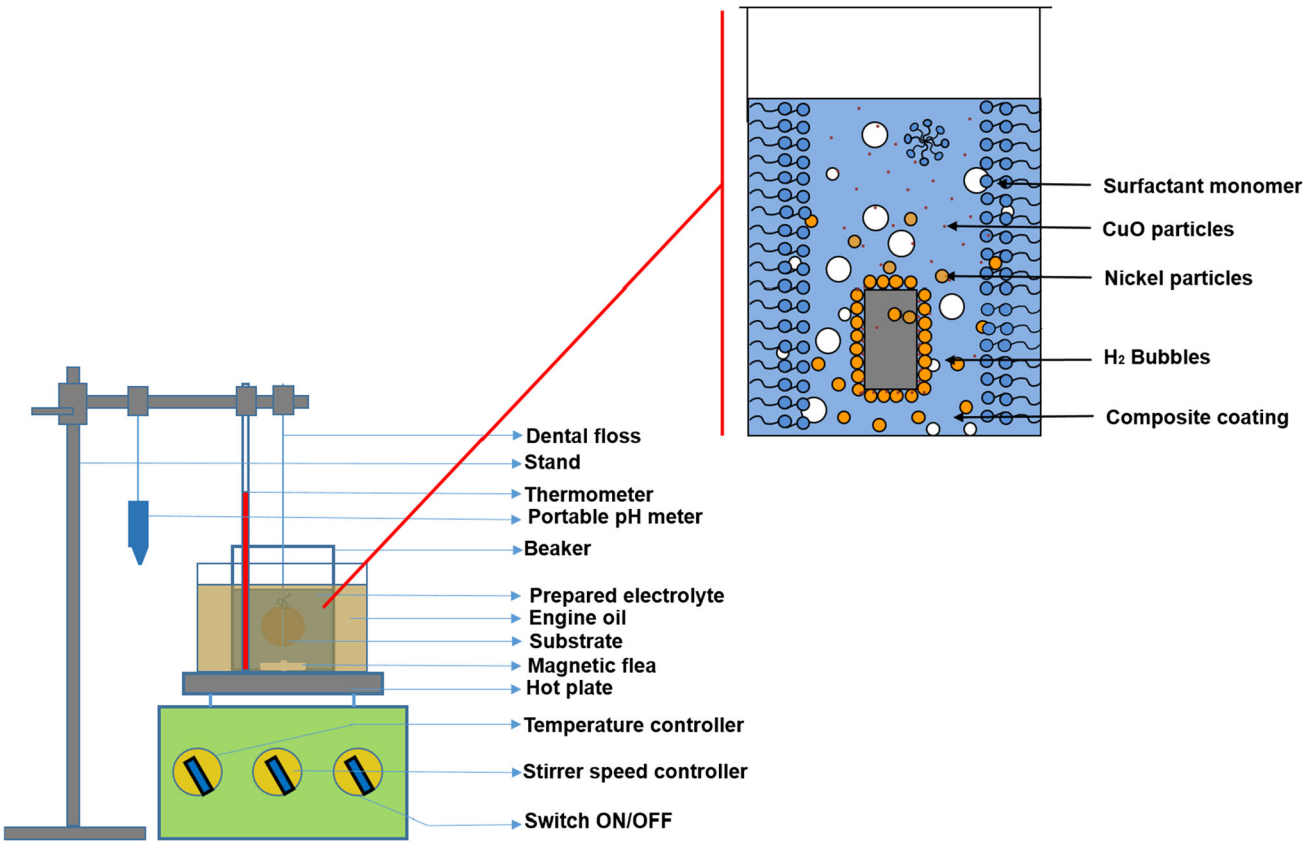

Fig. 2 Schematic representation of working of surfactant in electrolyte

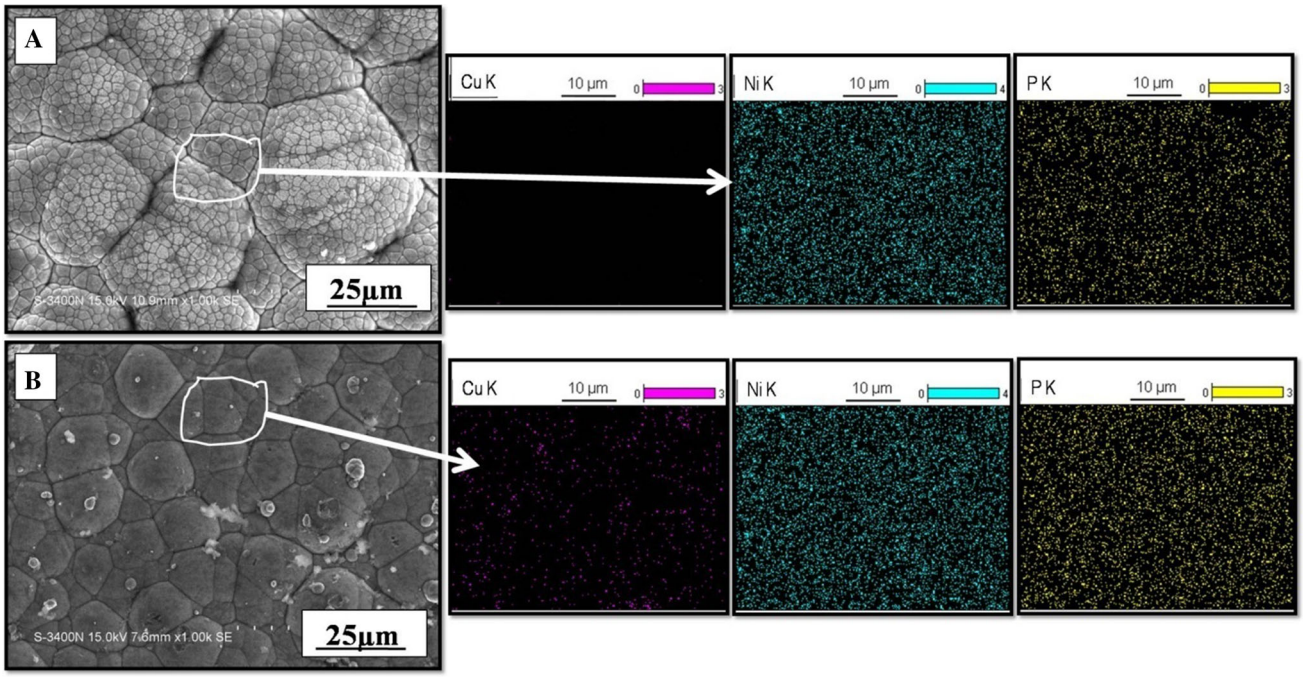

Fig. 3 SEM morphology of electroless **a** Ni–P **b** Ni–P–CuO coating on mild steel

Table 2 Composition of deposits determined by EDS analysis

Type of coating	Ni (wt.%)	P (wt.%)	Cu (wt.%)
Ni–P	94.3	5.6	0
Ni–P–CuO	92.3	7.4	0.3

pared electrolyte, the nanoparticles were not agglomerated with each other. Mechanical stirrer facilitated the nanoparticles to float throughout the coating process. Figure 2 shows the schematic representation of electroless composite coating setup and the working of Zwitterionic surfactant in electrolyte during coating process. These could be the possible reasons for improving the deposition rate and nanoparticle suspension in the electrolyte during the coating process. In addition, Zwitterionic surfactant is applicable for all composite coatings to get the similar effect in electroless coating process.

3.2 Characterization of Composite Coating

The SEM micrographs of electroless coated substrates are indicated in Fig. 3. The surface of the deposit was compact which displays a nodular form in Ni–P matrix as shown in Fig. 3a. In addition, it shows the mapping of Ni and P content. The presence of Cu was not observed in the deposit. Copper co-deposition has played a major role in reducing nodule sites, refinement in microstructure is clearly visible and the presence of nano-sized particles as indicated in Fig. 3b. The Ni–P and Ni–P–CuO coatings are formed by autocatalytic reaction and the precipitates of CuO co-deposited in Ni–P to make the adherent Ni–P–CuO coating. Electroless coated Ni–P and Ni–P–CuO deposit elements determined by EDS analysis are shown in Table 2.

Binary Ni–P deposit contains 92.3 wt.% Ni and 7.6 wt.% P. Co-deposition of copper oxide resulted in composite Ni–P alloy with the copper content of 0.3 wt.% and phosphorus content of 7.4 wt.%. Similarly, it was observed that there was no decrease in phosphorus content as a result of the incorporation of copper in Ni–P deposit in the form of nano-copper oxide. The X-ray diffraction patterns of the coated with Ni–P and Ni–P–CuO deposits are shown in Fig. 4a, b. The reflections corresponding to the (111) plane of a face centre cubic phase of nickel could be observed from the analysis. From Fig. 4b it is evident that as-deposited binary Ni–P has three most intense peaks of the pattern indexed as (111), (220) and (222) reflections of the nickel phase. The presence of co-deposited copper reflects the peak at an angle of 38.78° shown in Fig. 4a, which is similar to trend that reported for Ni–P coatings. The Ni–P deposit shows the highly crystalline structure whereas in the case of Ni–P–CuO produces the amorphous structure. Literature reports that electroless Ni–P

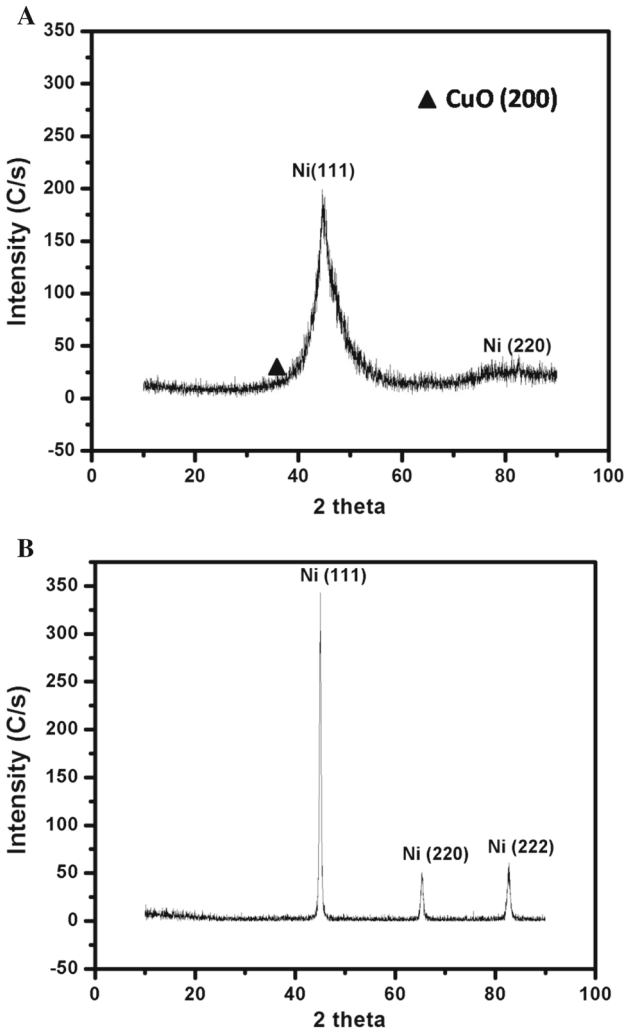

Fig. 4 XRD patterns of electroless coatings. **a** Ni–P–CuO **b** Ni–P

deposits possess 1–3 wt.% crystalline P and 6–8 wt.% amorphous P [19]. The CuO peak was merged with the amorphous since the CuO content in the deposit was comparatively very low (only 1%) and the scan rate was limited where CuO peaks were observed.

3.3 Surface Properties of Prepared Composite Coating

The average surface roughness (R_a) of the deposits as measured and the evolution profiles are represented in Fig. 5. High R_a value was obtained on Ni–P deposit (2.29 µm) due to the presence of high nickel content (94.3 wt.%). These values followed an analogous trend reported by earlier researchers [20,21]. The R_a value of Ni–P–CuO was 1.956 µm. Co-deposition of copper in the deposit appears to stifle the development of the nodules by inhibiting further development. This could be the principle reason for getting smooth deposits of composite Ni–P–CuO when compared

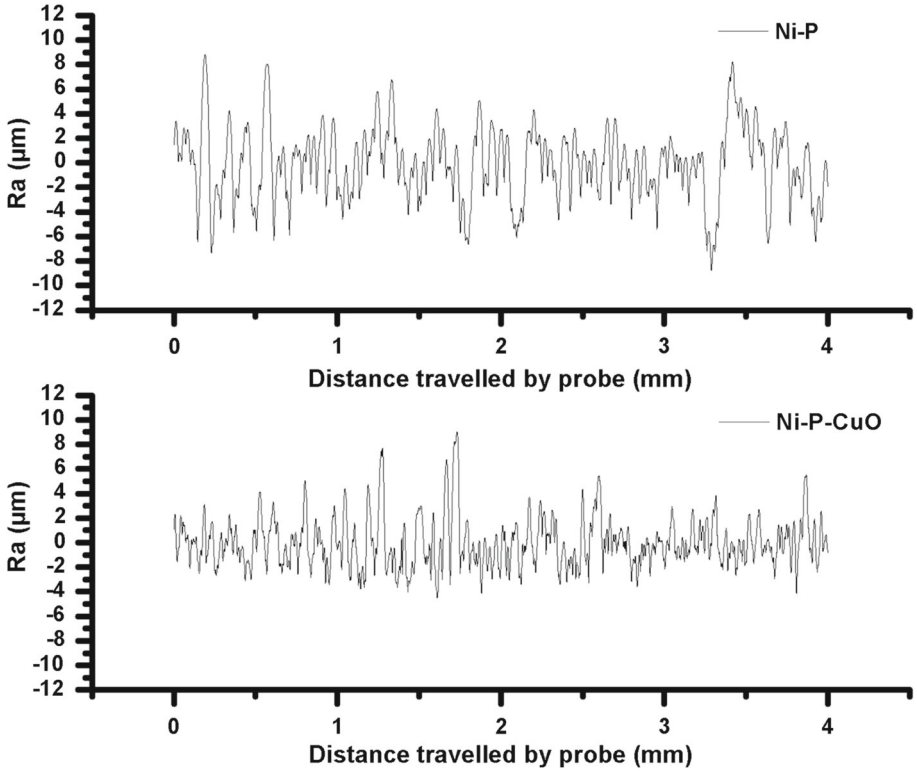

Fig. 5 Evaluation profile of electroless coated deposits for roughness measurement

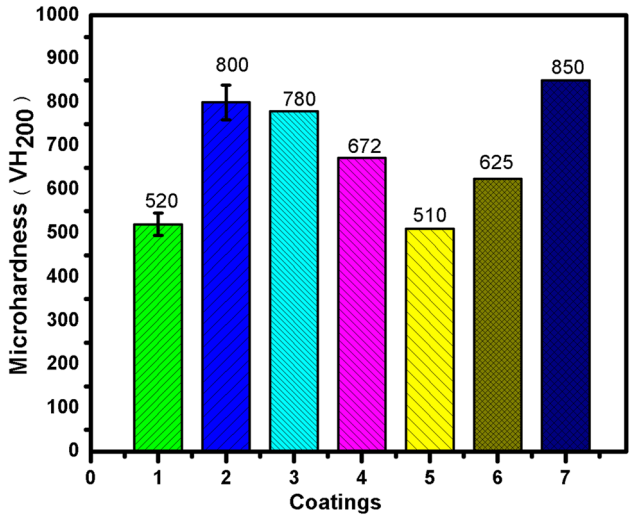

Fig. 6 Comparison of microhardness for different electroless coated deposits. (1) Ni–P, (2) Ni–P–CuO, (3) Ni–P-CNT, (4) Ni–P–Ag, (5) Ni–P–SiC, (6) Ni–P–TiO_2 and (7) Ni–P–Al_2O_3 [8–11]

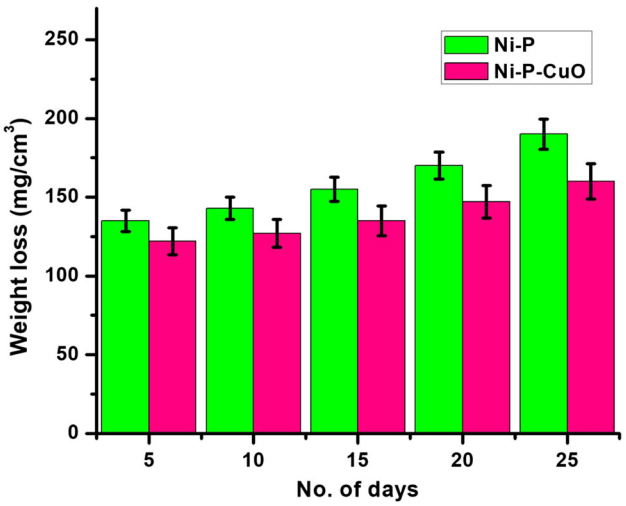

Fig. 7 The weight loss of the substrate subjected to immersion corrosion test

to the substrate without nano-copper. The copper was added by 1% only; hence, it does not affect the surface roughness. The substrates coated with and without copper nanoparticles were tested for microhardness. The binary Ni–P deposit produced the microhardness value of 580 HV_{200}, the deposit which has reinforcement produced 800 HV_{200}. Similarly, the earlier researcher tried with different hard nanoparticles such as CNT, Ag, SiC, TiO_2, Al_2O_3 and the corresponding microhardness values were 780, 672, 510, 625, 850 HV_{200}, which are shown and compared in Fig. 6.

Among these nanoparticles, Al_2O_3 produced the maximum hardness value followed by CuO which produced 800 HV_{200}. Recent researches reported that the electroless Ni–B, Ni–B–TiO_2, Ni–B–SiO_2, Ni–B–CuO and Ni–B–Al_2O_3 composite coatings showed the microhardness values of 270, 280, 290, 300 and 390 HV_{100}, respectively [22]. High microhardness was obtained due to hard particles (compared to nickel)

which were inserted in between the nickel particles to resist the load/force on the substrate. Moreover, the uniform distribution of CuO particles in the deposit resulted in greater resistance to plastic deformation [23]. The weight loss of binary alloy deposit and composite deposit increased gradually with respect to time as shown in Fig. 7. It was observed that the corrosion resistance of composite coating was better than Ni–P coating. The mild steel material was corroded easily with high weight loss [21], whereas the Ni–P-coated substrate showed good resistance to corrosion and weight loss were gradually increased. After the immersion test, the coated substrates were covered with deposit and the mild steel substrates were corroded severely. It is concluded that the substrate produced with Ni–P–CuO improved the corrosion resistance of the material.

4 Conclusions

- Electroless coating with CuO particles was obtained successfully using electroless coating process.
- Zwitterionic surfactant enhanced the deposition rate of composite coating and assisted in distributing the nanoparticle effectively.
- R_a value of the deposit decreased after addition of copper into the Ni–P matrix.
- CuO particles have significantly improved microhardness to 800 HV_{200} when compared to the binary-coated substrates (520 HV_{200}). No agglomeration of nanoparticles was seen during the coating process.
- The corrosion resistance of the substrate was improved significantly using Ni–P–CuO deposit.
- No decomposition of electrolyte was observed during the coating process. This will oblige all the electroless plating industries to make use of Zwitterionic surfactant when the composite coatings are prepared.

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