

Substrate pretreatment using plasma etching to enhance electroless Ni-P coatings performance

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Traditional electroless Ni-P films are produced using a substrate pretreatment (sensitization and activation) and chemical plating. The Taguchi method is used to determine the optimal coating parameters for electroless Ni-P films to achieve the best hardness and resistance to fatigue. The films are characterized using X-ray diffraction, scanning electron microscopy, by determining the fatigue life and using a nanoindenter. Analysis of variance results for the fatigue life for Ni-P films, the deposition time, the bath temperature and the pH value of the solution have respective contributions of 64.61%, 19.34% and 13.17%. In the confirmation tests, the respective values for fatigue life and the films' hardness increase from 42.25 times and 548.9 HV to 47.67 times and 572.4 HV. This study replaces a traditional substrate pretreatment with oxygen plasma etching. Oxygen plasma etching changes the morphology of the substrate's surface, increases the surface free energy and produces slight pitting and micro roughness to give a surface that is more hydrophilic. Substrates that undergo pretreatment using oxygen plasma etching have enhanced mechanical properties because a relatively good Ni-P film is produced. The results show that the fatigue life is increased from 47.67 times to 63.33 times and the Ni-P film's hardness is increased from 572.4 HV to 623.8 HV.

Keywords: electroless plating, Taguchi methodology, oxygen plasma etching, mechanical property.

1. INTRODUCTION

Brass is an alloy (Cu-Zn) that finds many applications in industry because of its superior electrical and thermal conductivity. However, the wear resistance of brass is poor and its surface is easily deformed and scratched, particularly at high temperatures [1], so its practical applications in industry are

limited [2]. Brass alloys are used for springs and bearings in mechanical parts that are subject to cyclic loading, which leads to fatigue failure [3]. Therefore, the fatigue strength and the wear and abrasion resistance of brass alloys must be increased [4]. Electroless nickel phosphorus (Ni-P) alloy is used as a protective coating for brass. It exhibits good mechanical and wear-resistance properties and the process for its deposition is easy and cheap [5]. Electroless plating uses an autocatalytic chemical reduction process that deposits metal onto both metallic and non-metallic surfaces.

There are many applications for electroless plating because it can be used to grow on complicated component surfaces and on many substrates, such as brass, steel, silicon, glass and ceramics, and it gives good resistance to corrosion and superior wear resistance [6]. Electroless coatings are used on various substrates for different purposes. Brass is one of the most extensively used metal alloys for electroless plating because it has many practical applications [7].

Subramanian et al. [8] reported the deposition of TiN coatings on copper and brass substrates with electroless nickel buffer layers. The adhesive strength and wear properties were determined using a scratch tester. Xu et al. [9] produced electroless Ni-P coatings on a brass substrate. The corrosion properties of the samples were determined using electrochemical impedance spectroscopy and a potentiodynamic polarization test. He et al. [10] coated Cu-Sn-Zn ternary alloy electroless plating films onto a brass substrate. The characteristics of ternary films are obviously superior to those of brass and there is a significant improvement in resistance to corrosion.

The Taguchi method combines mathematical and statistical techniques to determine the solutions for complex problems that involve a large number of variables using relatively few actual experiments [11]. It optimizes the prediction process using an orthogonal array and a signal-to-noise (S/N) ratio and an analysis of variance (ANOVA) [12]. The Taguchi method is suited to the study of film coatings, because the properties of a film are affected by many coating parameters, which can be optimized to save on labor and cost and to improve quality [13]. Sahoo [14] reported the wear characteristics of electroless Ni-P coatings sliding against steel using a Taguchi orthogonal design. The results show that the annealing temperature and the bath temperature have the most significant effect on the wear properties of electroless Ni-P film coatings. Shu et al. [15] reported the optimization of the plating parameters for an electroless Ni-W-P film coating using the Taguchi method with an orthogonal array. The results show that the percentage of W and P in the ternary films can be optimized by controlling the bath composition and the process parameters.

This study uses a Taguchi design of experiment to fabricate electroless Ni-P alloy films on brass substrates. The S/N ratio is used to determine the best level for the parameters for electroless plating and an ANOVA is used to determine the significance of each parameter. The effect of the coating process on the structure, the surface morphology, the hardness and the fatigue life of the electroless Ni-P alloy films is reported. The effect on electroless Ni-P plating is also compared for a traditional pretreatment (sensitization and activation) and physical plasma etching.

2. EXPERIMENTAL

The experiments used a brass sheet of 0.5 mm in thickness (C2600, Ni: 1.82%, Si: 0.75%, Zn: 0.01%, Sn: 0.37%, Cu: balance). The specimen was progressively mechanically polished using 100 to

1200 grit papers, to avoid residual stresses. Substrates were cleaned to remove chemical impurities and any contamination using a standard Radio Corporation of American (RCA) cleaning process. Fig. 1 shows the shape of the brass sample, the middle zone (thickness is 0.5 mm) for the electroless Ni–P coating and the fatigue test.

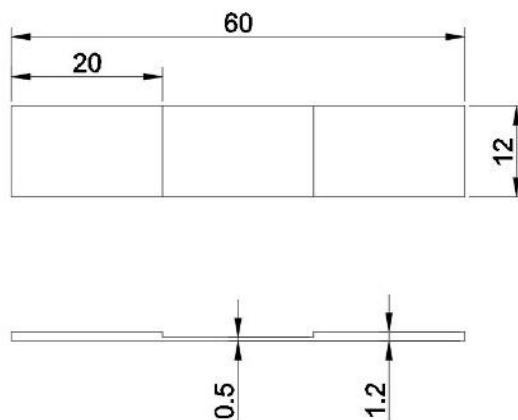


Figure 1. Shape of the brass sample, the middle zone (thickness is 0.5 mm) for the electroless Ni–P coating and the fatigue test: all dimensions in mm.

For comparison purposes, traditional and the proposed substrate pretreatments were applied before the electroless coating process. Fixed compositions were used for the electroless plating bath [NiSO₄ (20 g/L), NaH₂PO₂ · H₂O (20 g/L), Na₃C₆H₅O₇ (34 g/L) and NH₄Cl (25 g/L)] to determine the effect of the deposition parameters for the electroless Ni–P film on the structure, fatigue life and micro hardness. A Taguchi experimental design used an L₉ (3⁴, with three columns and nine rows) orthogonal array. Table 1 shows the results for substrates that are pretreated using traditional and physical methods and the bath composition and deposition conditions for electroless Ni–P plating. The pH of the bath is one of the factors that significantly affect the P content in the electroless Ni–P coating.

Table 1. Results for substrates that are pretreated using traditional and physical methods and the bath composition and deposition conditions for electroless Ni–P plating.

Substrate	Brass (specimen shape see Figure 1)			
Stirring rate	350 rpm			
Traditional substrate pretreatment:				
Sensitization (H ₂ CrO ₄ 150 g/L) + (H ₂ SO ₄ 10 g/L), duration time 2 min				
Activation (HCl 230 g/L), duration time 2 min				
Electroless plating bath composition	NiSO ₄	20 g/L		
	NaH ₂ PO ₂ · H ₂ O	20 g/L		
	Na ₃ C ₆ H ₅ O ₇	35 g/L		
	NH ₄ Cl	25 g/L		
Symbol	Control factor	Level 1	Level 2	Level 3
A	solution pH value	8	9	10
B	bath temperature (°C)	75	85	95

C deposition time (min)	10	15	20
Proposed substrate pretreatment: Oxygen plasma etching parameters: base pressure 2.5×10^{-5} Torr, frequency 40 kHz, oxygen flow rates 60 sccm, direct current power 230 Watt, duration time 8, 12 and 16 min.			
The amount of phosphorous in the Ni–P alloy is governed by the following reaction [16]: $2\text{H}_2\text{PO}_2^- + \text{H}^+ \rightarrow \text{P} + \text{H}_2\text{PO}_3^- + \text{H} + \text{H}_2\text{O}$ (1)			

The phosphorus content decreases as the solution pH increases. Hsu et al. [17] used the Taguchi method to determine the effect of the parameters for electroless Ni–P coating on the structure, mechanical properties and fatigue strength. The optimal parameters for an electroless Ni–P film coating are a solution pH of 4.0, a bath temperature of 70°C and a deposition time of 60 min. The coating of electroless Ni–P films is sensitive to the solution pH. This study uses a higher pH value (alkaline solution pH = 8, 9 and 10) and shorter deposition time (10, 15 and 20 min) to determine the effect of control factors on the electroless Ni–P coating films.

The surface morphology and cross-sections of the specimens were determined using a field emission scanning electron microscope (SEM, JEOL JSM-6500F). The phase structure of the film was identified using X-ray diffraction (Rigaku-2000 X-ray Generator), with Cu K α radiation that is generated at 40 kV and 40 mA. The angle of incidence was adjusted to 2°, in order to get information from the near surface. Energy dispersive X-ray spectroscopy (EDS) was used to determine the semi-quantitative chemical composition of the films. The thickness was measured using a surface profilometer (α -step, AMBIOS XP-1). The micro-hardness of the films was measured at room temperature using a nano-indenter (Mitutoyo HM-100 Series). The low cycle fatigue life tests (strain is fully reversed: called the strain-life method) account for elastic and plastic behavior in localized regions where cracks initiate [18]. The fatigue life was measured by repeated bending. A bending angle of 50° and a completely reversed stress cycle were used for all fatigue tests. This study uses a stress ratio of R = -1, which is the ratio of the compressive stress to the tensile stress and is similar to the method of Hsu et al. [17] and Kuo et al. [19].

2.1. Analysis of the S/N ratio

For this study, the fatigue life is a higher-the-better (HB) category of performance characteristics for the Taguchi method. The S/N ratios for HB are calculated as:

$$\text{HB (maximize): } S/N = -10 \log \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right] \quad (2)$$

where n is the number of observations and y is the measured data: the S/N ratio for the HB or LB characteristic (unit: dB).

2.2. Analysis of variance (ANOVA)

An ANOVA is used to determine which deposition conditions for electroless Ni–P plating significantly affect the performance characteristics.

$$S_m = \frac{(\sum \eta_i)^2}{9}, \quad S_T = \sum \eta_i^2 - S_m \quad (3)$$

$$S_A = \frac{\sum \eta_{Ai}^2}{N} - S_m, \quad S_E = S_T - \sum S_A \quad (4)$$

$$V_A = \frac{S_A}{f_A}, \quad F_{Ao} = \frac{V_A}{V_E} \quad (5)$$

where S_T is the sum of squares due to the total variation, S_m is the sum of squares, S_A is the sum of squares due to a parameter A (A = solution pH value, bath temperature or deposition time), S_E is the sum of squares due to error, η_i is the η value for each experiment ($i = 1, 2, \dots, 9$) and η_{Ai} is the sum of the i^{th} level for parameter A ($i = 1, 2, 3$), N is the repeating number for each level of parameter A , f_A is the degree of freedom for parameter A and V_A is the variance for parameter A [20].

3. RESULTS AND DISCUSSION

3.1 Traditional substrate pretreatment using sensitization and then activation

Electroless Ni–P film plating involves surface sensitization, surface activation and chemical plating. The thickness of all of the electroless Ni–P films coatings that are used in this study is 1.35 ~2.20 μm . Table 2 lists the experimentally measured values for Vickers hardness for coated (317.6 ~ 548.9 HV) and uncoated electroless Ni–P plating (229.4 HV). Standard deviation (Stdev.) is used to measure confidence in statistical conclusions. The Vickers hardness measurement ranges from 3.1 to 7.5. The small deviation shows that the experimental results are close to the average value. All experiments were conducted four times.

Table 2. Vickers hardness for all electroless Ni–P plating samples.

Exp.	Factors			Vickers Hardness (HV)				Avg.	Stdev.
	A	B	C	Test 1	Test 2	Test 3	Test 4		
1	1	1	1	319.1	320.9	320.5	309.9	317.6	4.5
2	1	2	2	467.3	469.9	458.6	457.7	463.4	5.3
3	1	3	3	485.3	484.6	500.6	490.4	490.2	6.4
4	2	1	2	436.4	439.2	447.9	430.2	438.4	6.4
5	2	2	3	519.2	514.2	511.0	530.8	518.8	7.5
6	2	3	1	374.5	372.0	379.5	367.6	373.4	4.3
7	3	1	3	457.3	452.8	458.2	450.8	454.8	3.1
8	3	2	1	418.7	421.7	410.8	420.4	417.9	4.2
9	3	3	2	546.6	545.1	553.7	550.1	548.9	3.3
Uncoated brass				232.3	228.7	223.1	233.4	229.4	4.0

Table 3 shows the fatigue life [17, 18] and the corresponding S/N ratio for all electroless Ni–P plating samples. The S/N ratio is calculated using Eq. (2). For all electroless Ni–P plating samples (orthogonal arrays from No. 1 to No. 9), the fatigue life is 36.25 to 42.25 times. The Stdev. value for the fatigue life ranges from 0.4 to 1.1, which is a relatively low value. The highest value for fatigue life is 42.25 times and was obtained for sample $A_3B_3C_2$ (No.9: solution pH value of 10, bath temperature of

95°C and deposition time of 15 min). The fatigue life for uncoated brass substrates is 22.25 times. The experimental results show that an electroless Ni–P coating film on a brass substrate produces a significant increase in the Vickers hardness and the fatigue life. The Ni–P coating films protect the substrate and inhibit the formation of fatigue cracks on the surface of the side that is subject to tension [21], which increases the fatigue strength of the substrate. The experimental results show that an increase in hardness gives an increased fatigue life, as expected.

The microstructure of electroless Ni–P films varies with the P content in the film. Fig. 2 shows SEM micrographs and the EDS analysis for Ni–P film coatings for different sets of experimental conditions: (a) No. 3: solution pH value = 8, (b) No. 6: solution pH value= 9 and (c) No. 9: solution pH value= 10. For this study, the P content decreases from 3.69 wt.% to 0.70 wt.% and the pH of the plating solution increases from 8 A to 10. A similar result was obtained by Liu et al. [16]. The electroless Ni–P layers exhibit a typical spherical nodular structure and the grains become slightly larger as the solution pH is increased.

The ANOVA results for the fatigue life of Ni–P films are listed in Table 4. These correspond to Table 3. The table shows the contribution ratio of each parameter. The deposition time, the bath temperature and the solution pH have respective contributions to the performance in terms of the fatigue life of 64.61%, 19.34% and 13.17%.

Table 3. Experimentally measured values for fatigue life for electroless Ni–P plating and S/N ratios (Taguchi orthogonal array L₉ 3⁴).

Exp.	Factors			Fatigue life time (Number of bending)				Avg.	Stdev.	S/N (dB) higher-the-better
	A	B	C	Test 1	Test 2	Test 3	Test 4			
1	1	1	1	34	35	35	34	34.50	0.5	30.75
2	1	2	2	40	39	40	39	39.50	0.5	31.93
3	1	3	3	41	39	41	40	40.25	0.8	32.09
4	2	1	2	39	40	38	38	38.75	0.8	31.76
5	2	2	3	43	42	40	41	41.50	1.1	32.35
6	2	3	1	36	38	36	35	36.25	1.1	31.17
7	3	1	3	41	39	40	38	39.50	1.1	31.92
8	3	2	1	38	40	37	39	38.50	1.1	31.70
9	3	3	2	42	43	42	42	42.25	0.4	32.52
Uncoated brass				25	24	27	25	25.25	1.1	

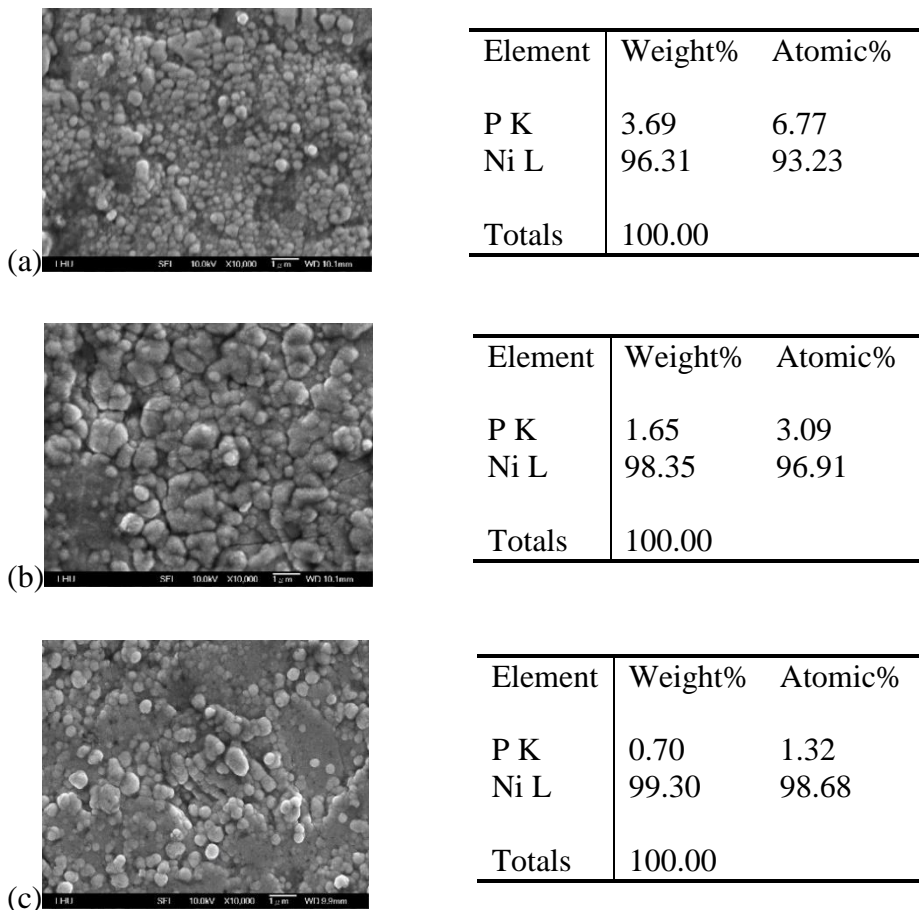


Figure 2. SEM micrographs and EDS analysis for Ni–P film coatings that are obtained using the experimental conditions for Nos. 3, 6 and 9 of the orthogonal array: (a) No. 3: solution pH value= 8, (b) No. 6: solution pH value= 9 and (c) No. 9: solution pH value= 10.

Table 4. ANOVA results for the fatigue life of Ni–P films.

Factors	Level (S/N)			Degrees of freedom	Sum of square	Variance	Contribution (P %)
	1	2	3				
A	31.59	31.76	32.04	2	0.32	0.16	13.17
B	31.48	31.99	31.93	2	0.47	0.24	19.34
C	31.21	32.07	32.12	2	1.57	0.79	64.61
Error	31.87	31.68	31.85	2	0.07	0.04	2.88
Total				8	2.43		100

The Taguchi method predicts that the greater the difference between the S/N ratios, the more significant is the effect on performance. The highest values - A₃ (solution pH value=10), B₂ (bath temperature= 85°C) and C₃ (deposition time= 20 min) - for each level 1–3 (Table 4) are the optimum conditions because the larger value for the S/N ratio, the closer is the result to the desired value. The

confirmation tests verify that the Ni–P film coatings show an improvement in the quality characteristic. The results for the confirmation experiment are compared with the results for the orthogonal array and the optimized parameters that are predicted by the Taguchi design and shown in Table 5. A comparison of the results for the optimal deposition parameters ($A_3B_2C_3$) and those for combination No.9 ($A_3B_3C_2$) shows that the fatigue life is increased from 42.25 times to 47.67 times and the films' hardness is increased from 548.9 HV to 572.4 HV. Yan et al. [22] reported that an electroless Ni–P coating with a low P content has a typical semi-crystallized structure (i.e. a mixture of amorphous phase and crystallized phase). Fig. 3 shows the X-ray diffraction patterns for the as-deposited Ni–P coatings. A sharp diffraction peak that is attributed to (111) of Ni is observed for the pure Ni deposit. A sharp Ni (111) diffraction peak indicates that the film is crystalline and exhibits a preferred orientation.

Table 5. Confirmation test for the optimal deposition parameters and those for combination No.9

	Orthogonal array $A_3B_3C_2$ (No.9)	Optimal parameters $A_3B_2C_3$	Improvement rate (%)
Fatigue life time	42.25	47.67	12.83
Vickers hardness (HV)	548.9	572.4	4.28

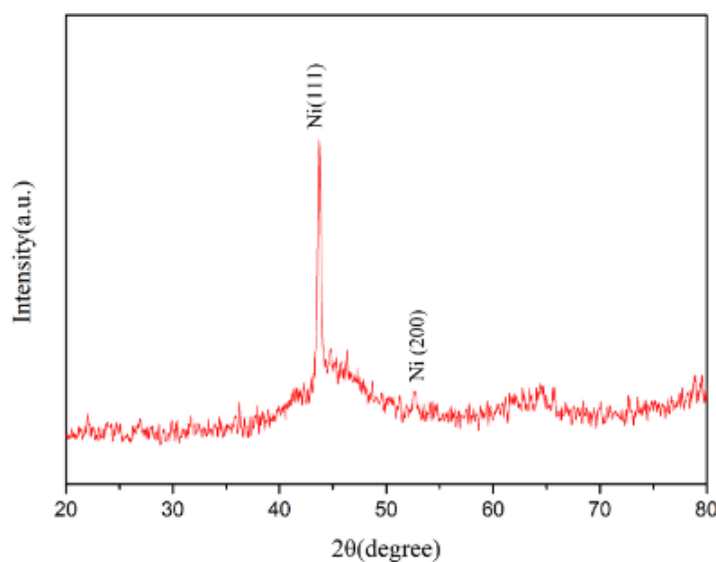


Figure 3. XRD patterns for the as-deposited Ni–P film.

3.2 Physical substrate pretreatment using plasma etching

Surface treatment using plasma technology is a non-contact and non-contaminating process that involves bombardment with energetic plasma. It changes the morphology of the substrate surface [23], increases the surface free energy and produces a surface that is more hydrophilic. Fig. 4 shows the SEM micrographs for the original surface (without pretreatment) and for brass substrates that are subject to different pretreatment regimes. In Fig. 4 (a), the original brass substrate shows slight cutting scratches

so this is not suitable for electroless coating. Fig. 4 (b) shows that the surface of the sample becomes rough after sensitization (H_2SO_4) and activation (HCl). The substrate surface is covered with a self-catalytic layer that renders the surface more chemically active so the Ni–P coatings adhere better to the surface [24]. After oxygen plasma etching for different periods (plasma etching conditions are shown in Table 1), there is slight pitting and micro-roughness on the surface (Figs. 4 c ~ e). As expected, after the pretreatment, the substrate surface becomes rough. The surface roughness slightly increases from $\text{Ra}=0.953 \mu\text{m}$ to $\text{Ra}=1.399 \mu\text{m}$.

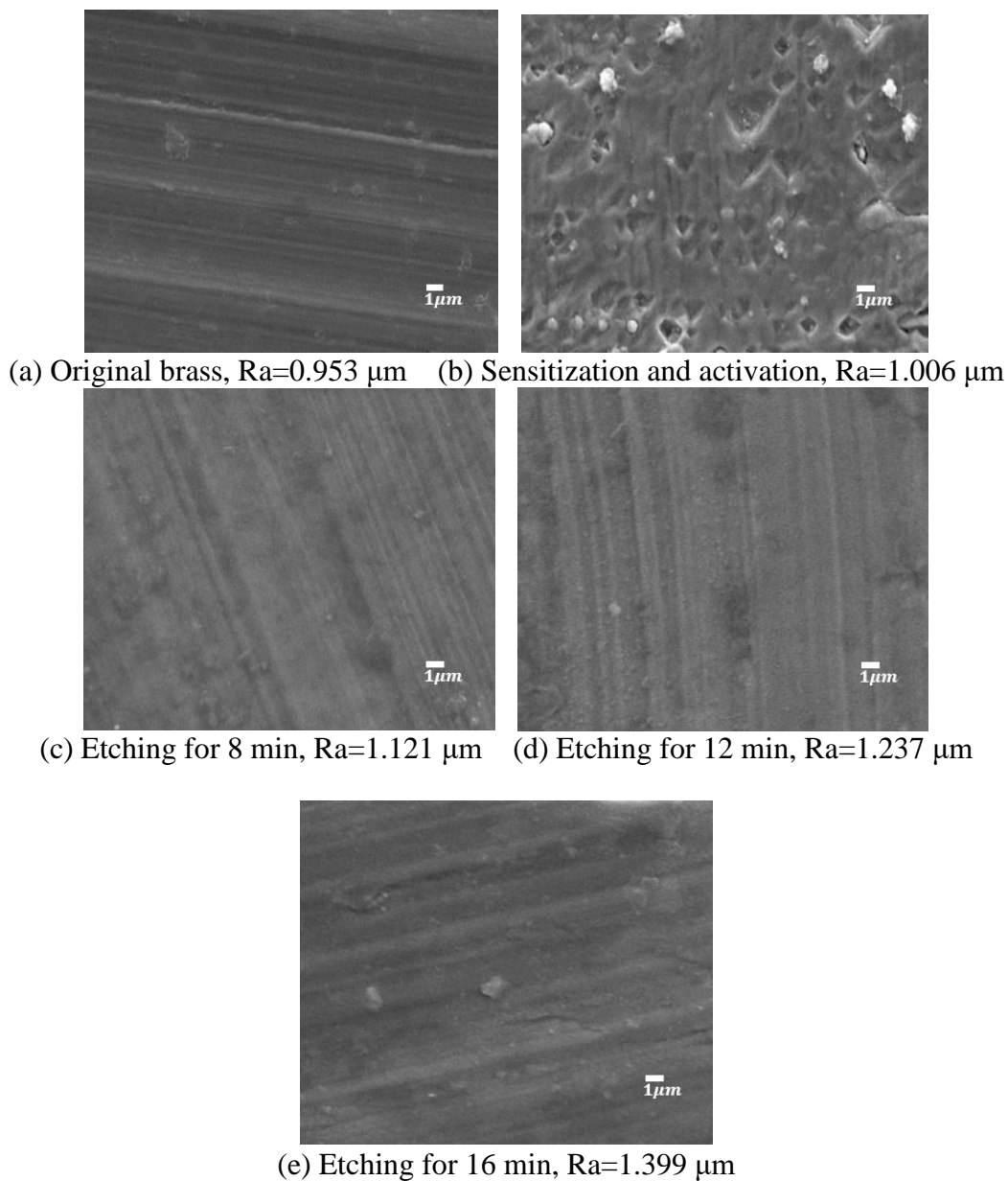


Figure 4. SEM micrographs of uncoated brass specimens: (a) without pretreatment, (b) traditional pretreatment, (c) the proposed pretreatment using a holding time of 8 min, (d) the proposed pretreatment using a holding time of 12 min and (e) the proposed pretreatment using a holding time of 16 min.

Fig. 5 shows the water contact angle for uncoated brass specimens, which corresponds to Fig. 4. In Fig. 5, the respective water contact angle for the original brass substrates is 90.7° and for those that undergo sensitization and then activation pretreatments the figure is 86.4° . For substrates that undergo oxygen plasma etching pretreatment for 8 min, the water contact angle is 83.7° and for those that undergo oxygen plasma etching for 16 min, the angle is decreased to 71.1° . Plasma etching improves wetting and surface modification enhances the polar component of the surface free energy of the substrate surface [25].

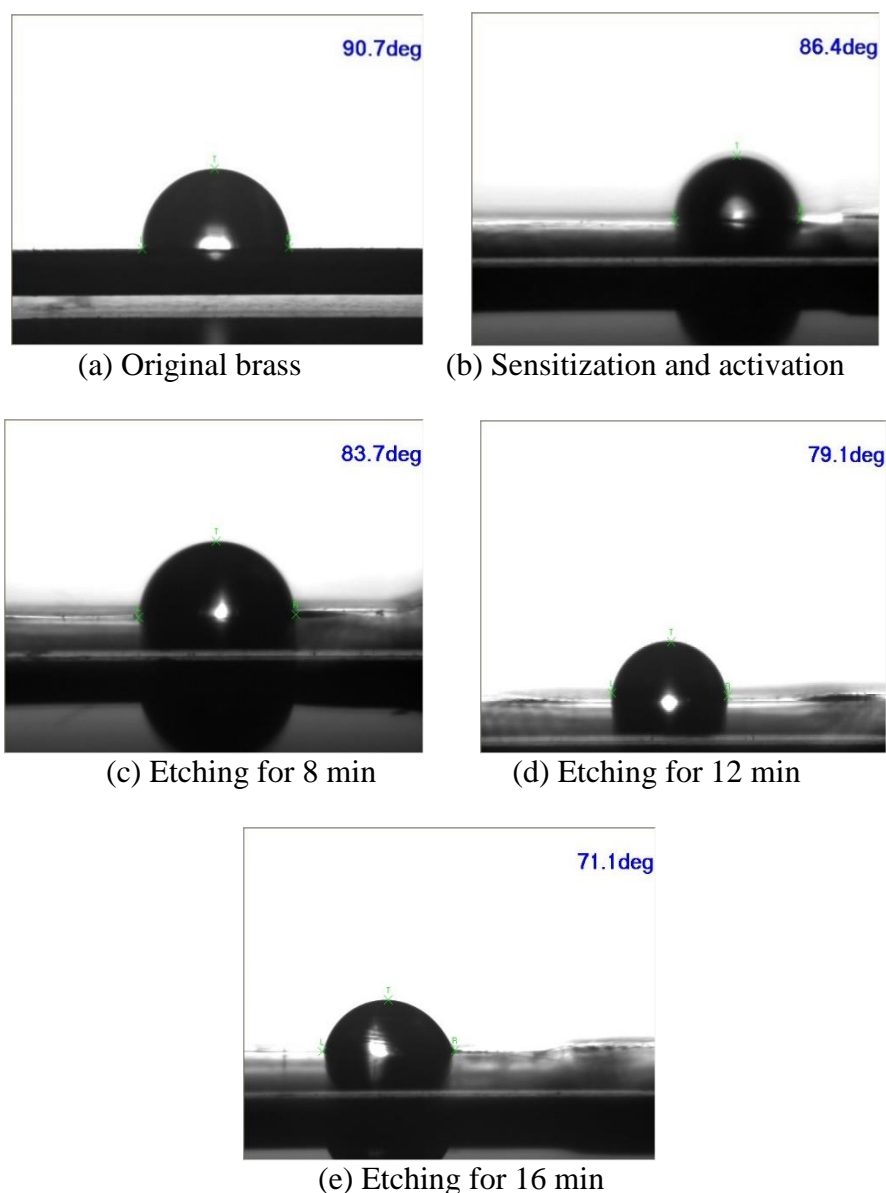


Figure 5. Water contact angle for uncoated brass specimens: (a) without pretreatment, (b) traditional pretreatment, (c) the proposed pretreatment using a holding time of 8 min, (d) the proposed pretreatment using a holding time of 12 min and (e) the proposed pretreatment using a holding time of 16 min.

Fig. 6 shows the SEM micrographs for an electroless Ni–P film that is deposited using the optimal deposition parameters ($A_3B_2C_3$) for different substrate pretreatments. The Ni–P film that is subject to traditional sensitization and activation (Fig. 6 a) has larger nodes and micro cracks or pores than those that undergo an oxygen plasma etching pretreatment (Figs. 6 b, c and d). The Ni–P film surface for substrates that undergo the proposed pretreatment is also smoother and more uniform than the surface of the substrate that undergoes a traditional pretreatment. Similar results were achieved by Liu et al. [26].

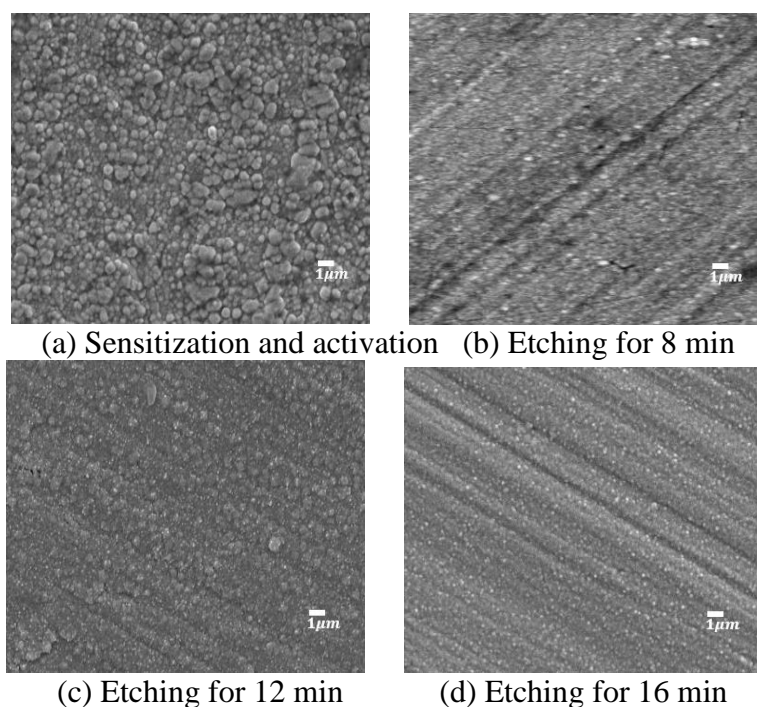


Figure 6. SEM micrographs for Ni–P film that is coated using the optimal deposition parameters ($A_3B_2C_3$): (a) traditional pretreatment, (b) the proposed pretreatment using a holding time of 8 min, (c) the proposed pretreatment using a holding time of 12 min and (d) the proposed pretreatment using a holding time of 16 min.

Table 6 lists the fatigue life and the Vickers hardness for Ni–P film that is coated using the optimal deposition parameters ($A_3B_2C_3$) for different pretreatments. The substrates that undergo pretreatment using plasma etching exhibit better Ni–P film characteristics. Substrates that undergo oxygen plasma etching pretreatment have better mechanical properties than those that undergo traditional sensitization and activation because a better Ni–P film is produced. A comparison between the results for the proposed pretreatment (holding time 16 min) and a traditional pretreatment show that the fatigue life is increased from 47.67 times to 63.33 times and the Ni–P film hardness is increased from 572.4 HV to 623.8 HV.

Table 6. Fatigue life and Vickers hardness for a Ni–P film that is coated using the optimal deposition parameters ($A_3B_2C_3$) for different pretreatments

	Fatigue life	Vickers hardness (HV)
Traditional pretreatment	47.67	572.4
Proposed pretreatment		
Holding time of 8 min	49.67	586.3
Holding time of 12 min	58.67	601.4
Holding time of 16 min	63.33	623.8

4. CONCLUSIONS

The Taguchi method and an ANOVA are used to determine the optimal parameter settings that increase the performance of electroless Ni–P coatings. A higher pH value (alkaline solution pH =8, 9 and 10) and a shorter deposition time (10, 15 and 20 min) are used to determine the effect of control factors on the electroless Ni–P coating films. Standard deviation is used to measure confidence in statistical conclusions. The experimental results show a small deviation so these are close to the expected value. The experimental results show that the use of an electroless Ni–P coating film significantly increases the Vickers hardness and the fatigue life. X-ray diffraction patterns for the as-deposited Ni–P coatings feature a sharp Ni (111) diffraction peak, which indicates that the film is crystalline and exhibits a preferred orientation. The confirmation tests show that the fatigue life increases by 12.83% and the hardness increases by 4.28, using the optimal parameters for the deposition of Ni–P film (solution pH =10, bath temperature= 85°C and deposition time= 20 min).

The effect of physical plasma etching on electroless Ni–P plating is studied. The Ni–P film surface for substrates that undergo the proposed pretreatment is smoother and more uniform than that for substrates that undergo a traditional pretreatment. The substrates that are pretreated using plasma etching exhibit better Ni–P film characteristics. The fatigue life is increased to 63.33 times and the Ni–P film hardness is increased to 623.8 HV.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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