




Effect of Chemical Additives in the Plating Bath on Surface Corrosion Resistance of Ni(P)

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In this work, the effect of the chemical additives (surfactant, stabilizer) on the corrosion resistance of the Ni(P) layer was investigated. The average O content at the depth of 1 nm of the tested Ni(P) specimens was used as the indication of the corrosion degree for the tested Ni(P) specimen. The average O content at the depth of 1 nm of the tested Ni(P) specimens prepared with thiourea is 27.74 at.%, which is larger than the average O content (14.43 at.%) at the depth of 1 nm of the corrosion-tested Ni(P) layer. It can be concluded that the corrosion resistance of the Ni(P) layer would be greatly reduced two times with adding thiourea in the Ni(P) plating bath. The effect of the solder mask was also investigated on the corrosion resistance of the Ni(P) layer. We found that S impurities leaching from the solder mask might be co-deposited into Ni(P), and decrease the corrosion resistance of the Ni(P) layer. Yet, its influence is relatively lower than the effect of thiourea on the corrosion resistance of the Ni(P) layer. With adding CTAB, as a surfactant, in the Ni(P) plating bath, the average P content in the Ni(P) layer increases. The high P content further improves the corrosion resistance of the Ni(P) layer. Moreover, the positive effect of adding CTAB in the plating solution on the corrosion resistance of the Ni(P) layer would suppress the negative effect of the thiourea additive and solder mask in the plating solution. The main innovation finding of the work is that we found that adding CTAB in the Ni(P) plating solution can reduce the co-deposition of S impurities, from thiourea in the Ni(P) plating bath and the solder mask, in the Ni(P) layer. Consequently, the corrosion resistance of the Ni(P) layer can be further improved.

Key words: Electroless nickel, thiourea, solder mask, corrosion, electron spectroscopy for chemical analysis (ESCA)

INTRODUCTION

Electroless Ni plating was invented by Brenner and Riddell in 1944.¹ Since then, it has been used in micro-electronic packing applications due to many advantages, such as, low-cost process, good wear-

resistance, excellent interfacial reaction with Pb-free solders, and so on.^{2–5} The key for the Ni(P) deposition process is the dehydrogenation reaction of the H_2PO_2^- in the plating bath. Accompanying the Ni deposition, the P content resulting from H_2PO_2^- would be co-deposited in the electroless Ni(P) layer. The electroless Ni(P) layer with a low P content (1–5 wt.%) is polycrystalline. The electroless Ni(P) layer with a medium P content (6–10 wt.%) has a mixing microstructure including polycrystalline and

amorphous. If the P content in the electroless Ni(P) layer is over 11–13 (wt.%), the microstructure of the electroless Ni(P) layer is mainly amorphous structure.⁶ It is known that the P content in the electroless Ni(P) layer would greatly influence the many properties of the electroless Ni(P) layer. The P content has a significant effect on the hardness, wear resistance, and the corrosion resistance of the electroless Ni(P) layer.⁷ The electroless Ni(P) layer often serves as a diffusion barrier for the metal bond pad in the electronic packaging. As the electroless Ni(P) layer reacts with Pb-free solders, a P-rich layer (Ni₃P phase) formed between Ni layer and intermetallic compound layer. The Ni₃P layer has been identified to weaken the interfacial strength.⁸

The stability of the temperature and pH value in the Ni(P) plating bath is very important. During the Ni(P) plating process, the possible local non-uniformity in the temperature and pH value in the Ni(P) plating solution could occur. As the instability situation occurs in the Ni(P) plating solution, the chemical sources in the plating solution tend to decompose in the Ni(P) plating process. Consequently, it causes the P content decreasing and fluctuating in the Ni(P) layer.^{9–11}

Hence, there are various additives that can be added into the electroless plating solution to stabilize and play certain functions in the plating solution for the Ni(P) deposition process. For example, thiourea can be added in the plating solution as the stabilizer for the stability of the chemicals.^{12,13} The surfactant is another important chemical additive added in the plating solution, which has a significant influence on the surface morphology, surface hardness, formation of low porous, and the reduction in H₂ gas bubbles adsorbed on the Ni surface during the plating reaction.¹⁴ Elansezhian et al.¹⁵ investigated sodium dodecylsulfate (SDS) and cetyl trimethyl ammonium bromide (CTAB) surfactant and showed a significant improvement in the surface finish roughness of the electroless Ni(P) layer on the steel surface.

We realized that the above chemical additives in the plating solution could be co-deposited in the Ni(P) layer, which could be the negative effect on the properties of the Ni(P) layer. Also, thiourea and the solder mask on the PCB could release S impurity in the Ni(P) plating bath, which also could be co-deposited with the Ni(P) deposition layer.^{16–18} And, eventually, the corrosion resistance of the Ni(P) layer would be damaged. Moreover, the interaction effect of those chemical additives (stabilizer, surfactant and S-contented molecules) in the plating solution on the corrosion resistance will be discussed in this paper.

EXPERIMENTAL PROCEDURE

In this work, a thin electroless Cu layer was first plated on 2-cm square blank printed circuit (PCB) boards as a seed layer. An electroplated Cu (2- μ m)

layer was electroplated on the thin electroless Cu layer. The surface of the electroplated Cu (2- μ m) layer was cleaned by a soft etching process with the 3,3'-Dithiobis-1-propanesulfonic acid (SPS) solution for 2 min. And, followed by 10 vol.% H₂SO₄ etching solution at room temperature for 1 min to remove the surface oxides on the Cu surface. Then, the clean Cu surface was activated by the Pd immersion process for the Ni(P) deposition. The Pd nucleated on the Cu surface serve as catalyst for the Ni(P) deposition, which adsorbs H₂PO₂ to enhance the dehydrogenation reaction rate. As a result, the Ni(P) electroless deposition process can be promoted.¹⁹

During the Ni(P) deposition process, several chemical agents, for example, NiSO₄·6H₂O, hexadecyl-trimethyl-ammonium bromide (CTAB), and thiourea, need to be added in the plating bath. NiSO₄·6H₂O serves as the Ni source and the sodium hypophosphite acts as the reducing agent in the Ni(P) plating bath. The stability of the temperature and pH value in the Ni(P) plating bath is very important. During the Ni(P) plating process, the possible local non-uniformity in the temperature and pH value in the Ni(P) plating solution could occur. As the instability situation occurs in the Ni(P) plating solution, the chemicals tend to decompose in the Ni(P) plating process. Consequently, it causes the P content decreasing and fluctuating in the Ni(P) layer. Thus, thiourea would be added in the plating solution as the stabilizer for stabilizing the chemicals. Also, hexadecyl-trimethyl-ammonium bromide (CTAB) has a long hydrocarbyl chain and the quaternary ammonium salt cation of CTAB can catch hydrophilic molecules. Therefore, CTAB functions as the surfactant, which can be adsorbed on the hydrophobic surface to remove these undesired molecules from Ni(P) surface. As a result, surfactant CTAB can effectively mitigate H₂ bubbles occurring on the plating Ni(P) surface. Hence, the formation of pores would eventually be eliminated significantly in the Ni(P) layer.

In the Ni(P) plating process, as Ni²⁺ in the plating solution is consuming, new metal sources would be added in the plating solution to replenish metal sources in the plating solution. The exhausted chemical additives (CTAB and thiourea) are considered as contamination and could be co-deposited with the Ni(P) deposition layer, which, eventually, damages the corrosion resistance of the Ni(P) layer.^{16–18}

In the work, we plan to investigate the effect of the possible contaminations (exhausted CTAB and thiourea and S-contained molecules) co-deposited with the Ni(P) deposition layer on the corrosion resistance of the Ni(P) layer. The pH value in the Ni(P) plating bath is controlled at 4.7. Different content of thiourea in the plating solution is controlled at 0 and 3 mg/L. Also, CTAB content in the plating solution is controlled at 5 mg/l.

After the completion of the Ni(P) plating process, the corrosion test was conducted by immersing the Ni(P) specimens in HNO₃ solution (volume ratio of HNO₃/H₂O is 1:1) for 10 s. After the corrosion test, DI water is used to clean the possible residual acid on the Ni(P) surface. The tested Ni(P) specimens were dried by N₂ gas for 10 s. ESCA depth analysis was used to investigate the chemical composition on the surface and along the different depth of the Ni(P) layer. The chemical binding energy of the ESCA spectrum would reveal the corrosion degree and the possible reactions between thiourea, CTAB, and S-contained molecules from the solder mask, which would greatly influence the corrosion resistance of the Ni(P) layer.

RESULTS AND DISCUSSION

Effect of Thiourea and Solder Mask on Corrosion Resistance of Ni(P)

During the HNO₃ corrosion test, the Ni(P) surface would be corroding and dissolving, which encourages oxidation forming on the corroding Ni(P) surface. With ESCA atomic depth profile results, the composition of various elements in different depths of the Ni(P) plating layer can be obtained. The discussion in this work mainly focus on the element distribution of O, P, and S, which are the potential contamination from the chemical additives in the Ni(P) plating solution. Besides the element distribution of O, P, and S, we will also include the analysis on the element distribution of C and N. The element distribution of C and N in the Ni(P) layer can be used to monitor how much the organic molecules in the Ni(P) plating solvent could be co-deposited into the Ni(P) layer.

Figure 1a shows ESCA atomic depth profile of the Ni(P) layer before the corrosion test. The unit of the x-axis of Fig. 1 is the depth-etching time in seconds. In this work, the depth-etching time on the studied Ni(P) layer was calibrated to be 0.5 Å/sec. This

calibration was done by measuring the etched thickness of the Ni(P) layer versus the etching time. In this work, we used the average O content at the depth of 1 nm (equivalent to the 20-s etching time in Fig. 1) of the corrosion-tested Ni(P) specimen to represent the corrosion degree of the corrosion-tested Ni(P) specimen. As shown in Fig. 1a, the average O content at the depth of 1 nm of the Ni(P) specimen before the corrosion test is 6.73 at.%. As shown in Fig. 1b, after the HNO₃ corrosion test, the average O content at the depth of 1 nm of the corrosion-tested Ni(P) specimen is 14.43 at.%. It is apparent that the average O content in the Ni(P) layer increases by 7.7 at.% after the HNO₃ corrosion test. Hence, after the corrosion test, the O content on the Ni(P) surface could be used as the indication of the corrosion degree in the tested Ni(P) specimen.

It is believed that thiourea (additive chemical) and solder mask could release S contamination in the plating bath during the Ni(P) plating process. We believe that the released S impurity in the Ni(P) plating bath would likely be co-deposited into the Ni(P) layer. The co-deposited S in the Ni(P) layer could possibly reduce the corrosion resistance of the Ni(P) layer. In the following, the effect of thiourea and solder mask on the corrosion resistance of Ni(P) are investigated and discussed. Firstly, we investigate the effect of thiourea alone on the corrosion resistance of the Ni(P) layer.

As shown in Fig. 2a and b, ESCA atomic depth analysis was also carried out on the corrosion-tested Ni(P) prepared with thiourea and solder mask, respectively. As shown in Fig. 2a, after HNO₃ corrosion test, the average O content at the depth of 1 nm of the corrosion-tested Ni(P) specimens prepared with thiourea is 27.74 at.%. Compared with the corrosion-tested Ni(P) specimens without thiourea (as shown in Fig. 1b), the average O content increases from 14.43% to 27.74 at.%. The increase in the average O content of the corrosion-tested Ni(P) specimens indicates that the Ni(P)

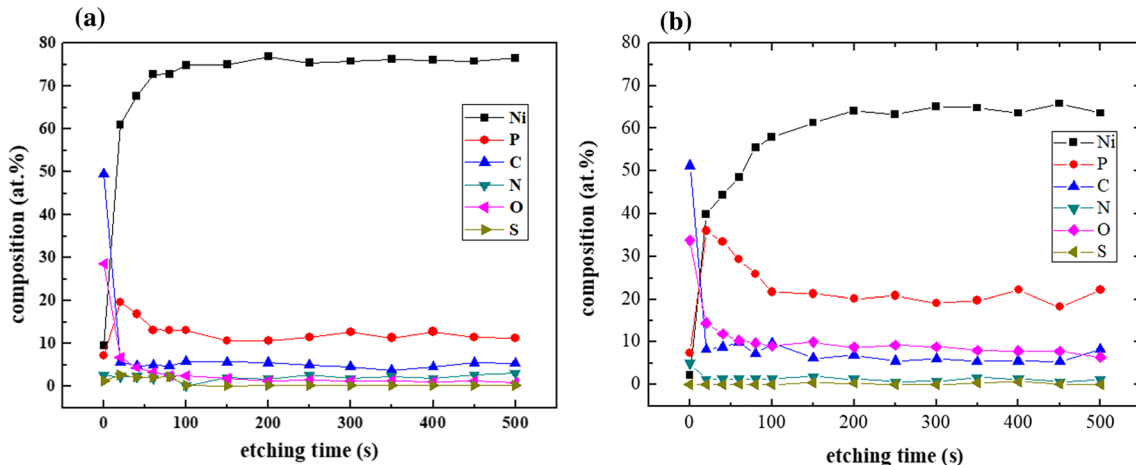


Fig. 1. ESCA atomic depth profile of the Ni(P) specimens for (a) Ni(P) specimens and (b) after HNO₃ corrosion test.

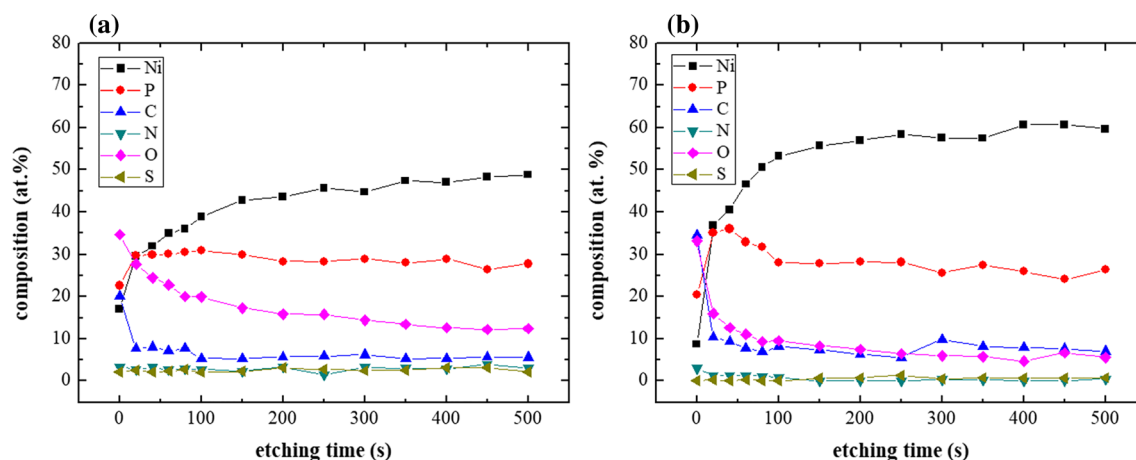


Fig. 2. ESCA atomic depth profile of the Ni(P) specimens after HNO_3 corrosion test prepared with adding different content of thiourea additive (a) without thiourea (b) with 3 mg/L thiourea.

layer prepared with thiourea would surely reduce the corrosion resistance of the Ni(P) layer.

How the thiourea additive reduces the corrosion resistance of the Ni(P) layer is not fully identified. The S content in the Ni(P) layer prepared with thiourea is too little to be detected by ESCA analysis. Yet, in the previous literature, Marcus et al. pointed out that the S impurity in the electroplated Ni and Ni-based alloys would reduce the metallic bonds between Ni atoms.¹⁹ Therefore, the co-deposited S impurity in the Ni(P) layer could enhance the corrosion and the dissolution rate of the Ni(P) layer.

This result is also confirmed by other studies.^{20,21} Thus, we tend to believe that, as thiourea is added into the Ni(P) plating bath, S is likely released from thiourea and could be co-deposited into the Ni(P) to decrease the corrosion resistance of the Ni(P) layer.

Photo-initiator and photo-sensitizer in the solder mask are another potential source for leaching S impurity in the Ni(P) plating bath. Thus, secondly, we investigate the effect of the solder mask on the corrosion resistance of the Ni(P) layer. As shown in Fig. 2b, after the HNO_3 corrosion test, the average O content at the depth of 1 nm of the tested Ni(P) specimens with solder mask on the PCB substrate is 16.01 at.%. Based on the results shown in Fig. 2b and 1b, we can conclude that, after the HNO_3 corrosion test, the average O content of the Ni(P) with solder mask increases. We assume that S impurities content leaching from the solder mask might be co-deposited into Ni(P), and affect the corrosion resistance of the Ni(P) layer. But, its influence is relatively lower than the effect of thiourea on the corrosion resistance of the Ni(P) layer.

Effect of CTAB on Corrosion Resistance of Ni(P)

Figure 3 shows ESCA atomic depth profile of the Ni(P) layer prepared with/without CTAB after the

corrosion test. As shown in Fig. 3a, the average O content of the corrosion-tested Ni(P) prepared without CTAB is 9.08 at.%. As shown in Fig. 3b, after the corrosion test, the average O content at the depth of 1 nm of the Ni(P) prepared with CTAB is 5.07 at.%. From the results above, we can understand that the Ni(P) layer prepared with CTAB can enhance the corrosion resistance of the Ni(P) layer. The possible reason is explained in below. It is known that the average P content can increase the corrosion resistance of the Ni(P) layer.¹⁵ Thus, to study the relation between the average P content of the Ni(P) layer and the corrosion resistance of the Ni(P) layer, we need to investigate the average P content of the Ni(P) layer prepared with and without CTAB (Fig. 4).

The average P content of the as-deposited Ni(P) layer prepared with and without CTAB contents can be estimated by ESCA analysis, we first estimate the average P content from the surface to the depth at 25 nm (equal to 500 s etching time) of the Ni(P) layer. With adding CTAB in the Ni(P) plating bath, we can see that the average P content in the Ni(P) layer increases from 11.8 at.% to 13.6 at.%. Therefore, we can confirm that the CTAB adding would increase the average P content in the Ni(P) layer, which further improves the corrosion resistance of the Ni(P) layer.

The possible enhancement mechanism of the corrosion resistance of the Ni(P) layer by increasing the P content is described below. As the corrosion occurring on the Ni(P) surface, the Ni would dissolve into the plating bath and P would be exposed out and form P oxides, i.e., hypophosphite (P^+) and phosphate (P^{5+}). These P oxides phases on the Ni(P) surface forming a passivation layer to inhibit Ni(P) from corrosion.²² Hence, the amount of P oxides, i.e., hypophosphite (P^+) and phosphate (P^{5+}), formed on the corroding Ni(P) surface would correspond to the corrosion resistance of the Ni(P) layer. The P_{2p} core-level peaks of the Ni(P) layer (in Fig. 5) were curve-

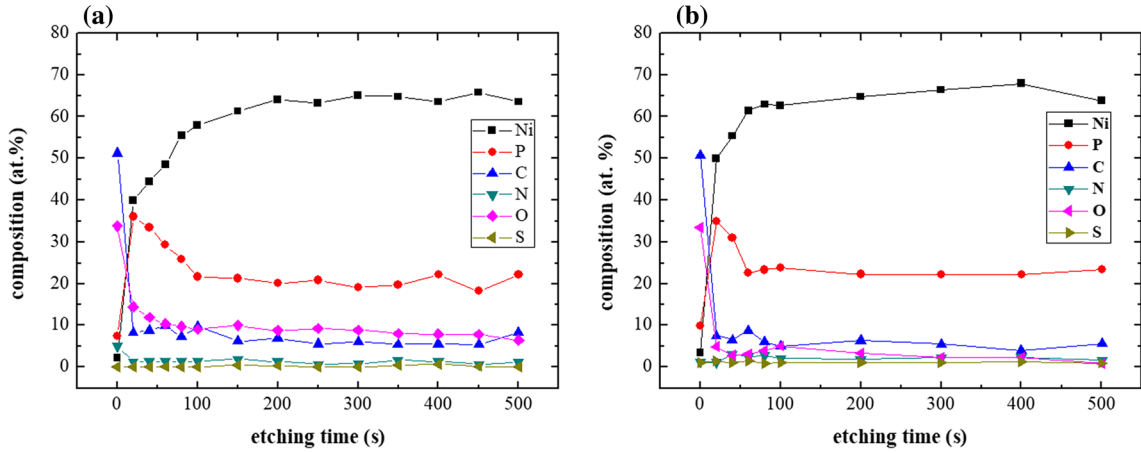


Fig. 3. ECSA atomic depth profile of the Ni(P) specimens after HNO₃ corrosion test prepared with adding different content of CTAB additive (a) without CTAB (b) with 5 mg/L CTAB.

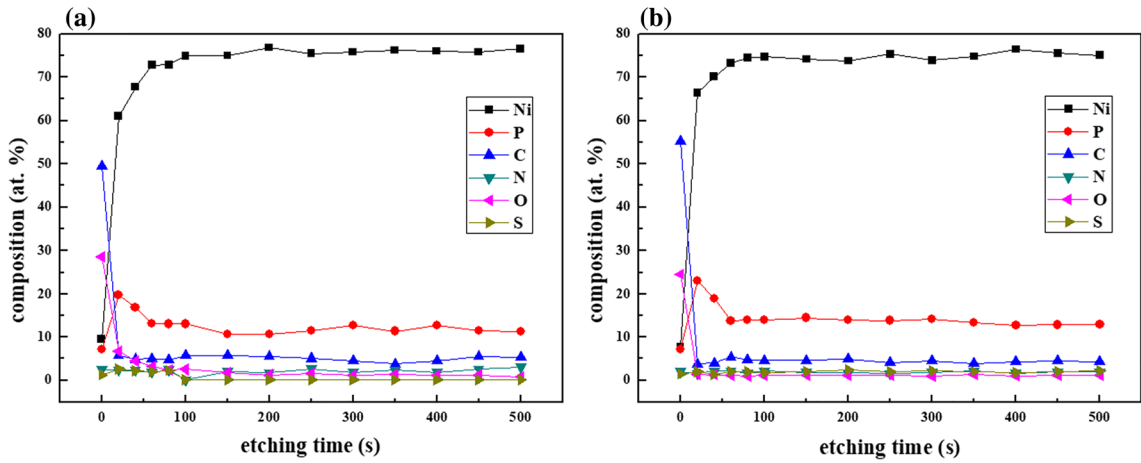


Fig. 4. ECSA atomic depth profile of the as-deposited Ni(P) specimens prepared with adding different content of CTAB additive (a) without CTAB (b) 5 mg/L CTAB.

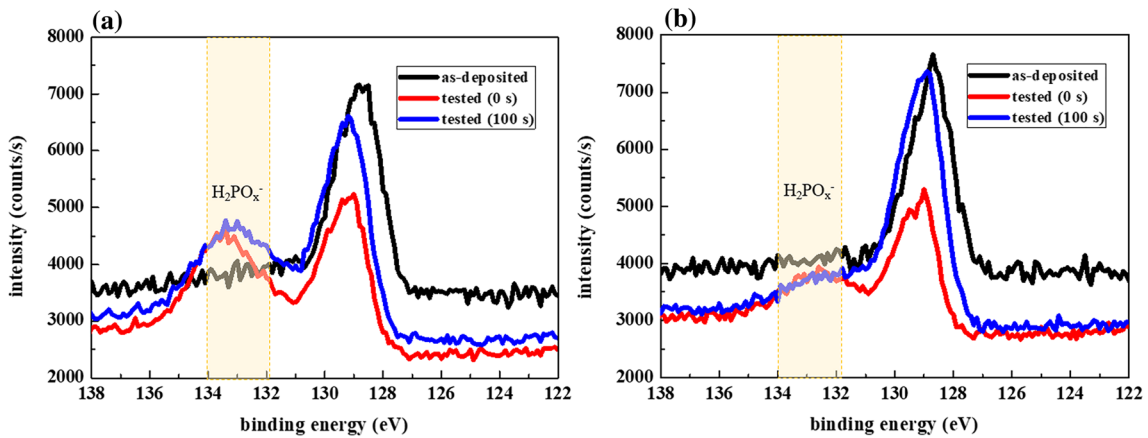


Fig. 5. ECSA for P_{2p} core-level peaks of the Ni(P) specimens after HNO₃ corrosion test prepared with adding different content of CTAB additive (a) without CTAB (b) 5 mg/L CTAB.

fitted into sub-curves shown in Fig. 5. The possible P oxides can be figured out by the sub-curves in the XPS spectrum in Fig. 5. As shown in Fig. 5a, the P_{2p} core-level peaks at 133.2 eV at 132.2 eV and 133.2 eV correspond to P^+ and P^{5+} of P oxides at the surface and at 5-nm depth of Ni(P), which can be used as the indication of the corrosion degree for the corrosion-tested Ni(P) specimen. As shown in Figs. 5, the intensity of the P_{2p} core-level peaks at 133.2 eV in the corrosion-tested Ni(P) prepared with CTAB is lower than that in the corrosion-tested Ni(P) prepared without CTAB. It again confirms that the CTAB adding would improve the corrosion resistance of the Ni(P) layer.

Figure 6 shows SEM images on the surface of the Ni(P) layer with or without CTAB. It is apparent that the amounts of cauliflower-like nodule and pinholes form on the surface of the Ni(P) layer prepared without adding CTAB. After adding CTAB, the surface of the Ni(P) layer is much smoother, and the amount of cauliflower-like nodule and pinhole were eliminated. During the plating process, the adsorbed hydrogen atoms would cause hydrogen bubbles on the plating Ni(P) surface. The H_2 on the surface of the Ni(P) has been identified as the root cause of the formation of pinhole or pits.¹⁶ CTAB can serve as surfactant to improve the wettability of the deposition layer on the Ni surface and lower the hydrophilic-lipophilic balance (HLB) values, which are the keys to decrease the interfacial tension between H_2 bubbles and the deposition layer and prevent H_2 bubbles absorbing on the plating Ni(P) surface. This is making H_2 bubbles that would not stay on the Ni(P) plating surface. As a result, the morphology of the Ni(P) surface would be much smoother, and improving the corrosion resistance of the Ni(P) layer.¹⁶

Interaction Effect of CTAB, Thiourea, and Solder Mask on Corrosion Resistance of Ni(P)

In the section (A), it has been concluded that both thiourea and solder mask would decrease the corrosion resistance of the Ni(P) layer. And, in the section (B), we also understand CTAB can enhance the corrosion resistance of the Ni(P) layer by increasing the P content in the Ni(P) layer. In this section, with adding CTAB in the plating bath, we will discuss how does the possible S impurities leached from both thiourea and solder mask co-deposited into Ni(P) layer affect the corrosion resistance of the Ni(P) layer, respectively.

Again, by analyzing ESCA depth profiles of the corrosion-tested Ni(P) with CTAB, thiourea, and solder mask as shown in Fig. 7, the composition of various elements in different depths of the corrosion-tested Ni(P) plating can be obtained. From the section (A), it can be concluded that, adding thiourea in the plating bath would decrease the corrosion resistance of the Ni(P) layer (see in Fig. 2a). Figure 2a shows the ESCA atomic depth profile of the corrosion-tested of Ni(P) plated with only thiourea additive, and the average O content at the depth of 1 nm is 27.74 at.%. As shown in Fig. 7a, the ESCA atomic depth profile of the corrosion-tested Ni(P) layer prepared with CTAB and thiourea additive together, the average O content is only 9.77 at.%. Clearly, we can see that the “positive” effect of CTAB is over the “negative” effect of thiourea on the corrosion resistance of the Ni(P) layer.

The ESCA atomic depth profile of the corrosion-tested of Ni(P) prepared with only solder mask (no thiourea) is shown in Fig. 2b. The average O content of the corrosion-tested Ni(P) prepared with solder

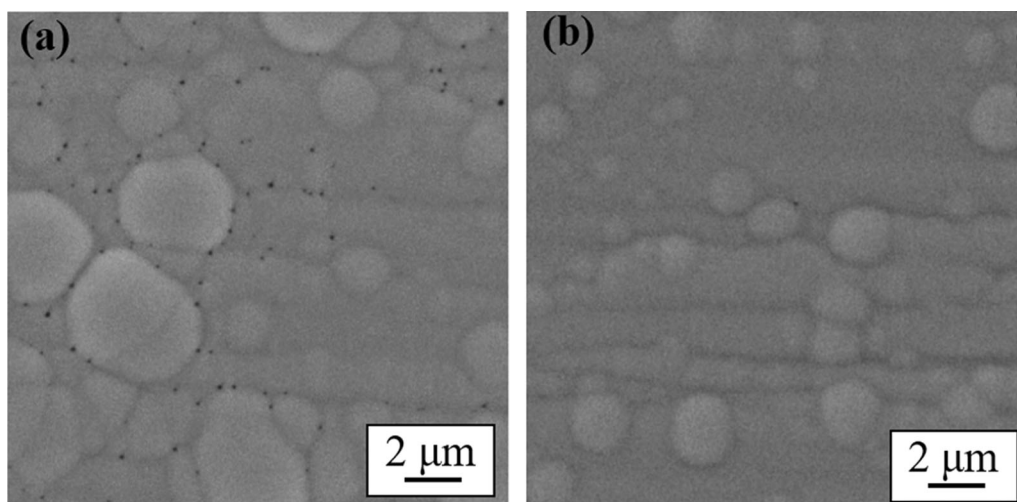


Fig. 6. SEM images of the as-deposited Ni(P) specimens prepared with adding different content of CTAB additive (a) without CTAB (b) 5 mg/L CTAB.

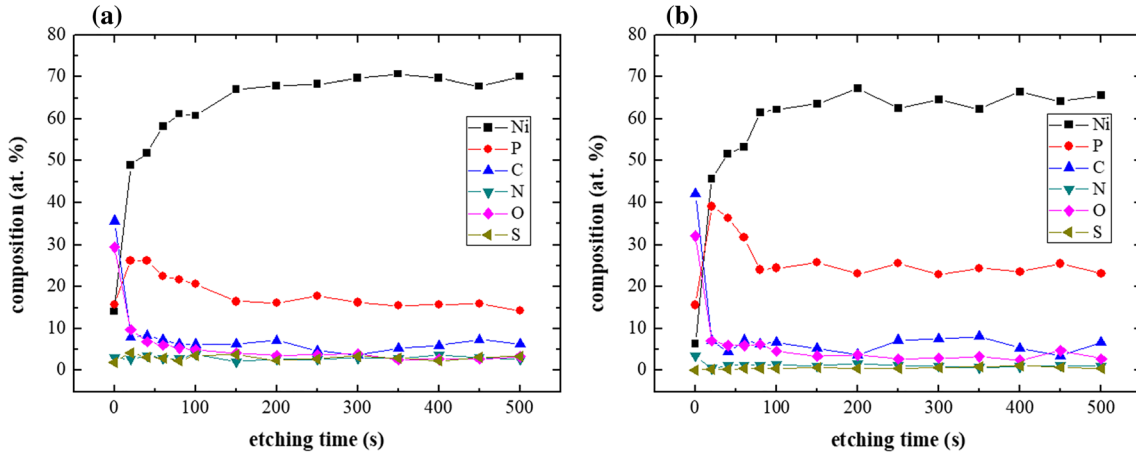


Fig. 7. ESCA atomic depth profile of the Ni(P) specimens after HNO_3 corrosion test prepared with adding 5 mg/L CTAB additive and (a) with 3 mg/L thiourea only (b) with solder mask only.

mask only (no thiourea) is calculated to be 16.01 at.%. As shown in Fig. 7b, for the corrosion-tested Ni(P) prepared with CTAB and solder mask together, the average O content at the depth of 1 nm of the corrosion-tested Ni(P) specimen is 7.10 at.%. Again, the “positive” effect of CTAB is over the “negative” effect of solder mask on the corrosion resistance of the Ni(P) layer. The mechanism of the “positive” effect of CTAB on the corrosion resistance of the Ni(P) layer is proposed below. During the Ni(P) plating process, CTAB would be adsorbed on the Ni(P) surface. The adsorbed CTAB molecules on the Ni(P) surface would reduce the probability of the S impurities (thiourea and solder mask) being adsorbed on the Ni(P) surface and co-deposited into the Ni(P) layer. Thus, the reduction of the S impurities co-deposited into the Ni(P) layer would likely improve the corrosion resistance of the Ni(P) layer.

CONCLUSION

With ESCA atomic depth profile results, the O content on the corrosion-tested Ni(P) surface would be used as the indication of the corrosion degree in the tested Ni(P) specimen. The average O content at the depth of 1 nm of the Ni(P) specimen before the corrosion test is 6.73 at.%. After the HNO_3 corrosion test, the average O content at the depth of 1 nm of the corrosion-tested Ni(P) specimen is 14.43 at.%. And, the average O content at the depth of 1 nm of the tested Ni(P) specimens prepared with thiourea is 27.74 at.%. It is apparent that the corrosion resistance of the Ni(P) layer would be greatly reduced with adding thiourea in the Ni(P) plating bath. We also investigated the effect of the solder mask on the corrosion resistance of the Ni(P) layer. We found that S impurities leaching from the solder mask might be co-deposited into Ni(P), and decrease the corrosion resistance of the Ni(P) layer. Yet, its influence is relatively lower than the effect of

thiourea on the corrosion resistance of the Ni(P) layer.

It is confirmed that the average P content in the Ni(P) layer would increase with adding CTAB, as a surfactant, in the Ni(P) plating bath. The high P content further improves the corrosion resistance of the Ni(P) layer. As discussed above, the positive effect of adding CTAB in the plating solution on the corrosion resistance of the Ni(P) layer would be over the negative effect of the thiourea additive and solder mask molecules in the plating solution.

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