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Electroless nickel-phosphorus plating on copper substrates by nickel strike activation

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GRAPHICAL ABSTRACT

process of plating copper substrate in KFJ-20 (EN).

ABSTRACT

Deposition of nickel layer onto copper substrate was investigated. The normal plating process which is cleaning, soft etching, pre-dipping, acid dipping and lastly electroless nickel plating have been performed onto copper substrates. No deposition of nickel layer onto the surface of copper surface when negative deposition speed -2.513 µm/h was obtained from our first trial. Nickel strike activation was introduced towards normal plating process in which nickel layers can be seen deposited on the surface of copper. The deposition speed of the nickel layer is 4.965 µm/h with semi-bright appearance. Optimization of the experimental parameters such as pH value, temperature and plating time were performed as they affected the deposition of nickel layer. The values for those parameters are 4.0-5.0, 90.0℃ and 20 mins, respectively. The optimum current density for nickel strike activation is 10 A/dm². The copper substrate was then characterized by scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) which showed the topography and chemical composition of the copper substrate. The result from these characterizations showed that copper substrates without nickel strike activation have irregular shape at several areas on the surface of copper and contain about 96.8 wt.% of copper, 2.8 wt.% of carbon and 0.4 wt.% of oxygen. While, for the copper substrate with nickel strike activation, discoidal shape of particle can be seen over the entire surface of the copper. The chemical composition of this copper substrate is a bit difference from the previous copper substrates where about 85.0 wt.% of nickel, 12.4 wt.% of phosphorus, 2.3 wt.% of carbon and 0.3 wt.% of oxygen were found in this substrate

Keyword: electroless nickel plating, copper substrate, nickel strike activation, deposition nickel layer

1. INTRODUCTION

Plating is a normal method being employed to cover the surface of metals. There are two types of plating which are electroplating and electroless plating. Electroplating is plating process that occurs with the flow of electricity while electroless plating is an autocatalytic plating method that involves several simultaneous reactions in an aqueous solution and occurs without electricity. Both methods of plating give the same main purpose which is to cover the surface of substrates but have different advantages.

Electroless plating is a chemical reduction process where its depends on catalytic reduction of a metallic ion from an aqueous solution, and the subsequent deposition of the metal without the use of electrical energy [1]. Electroless plating is popular in the world of electronic device such as hybrid circuits, multichip modules, capacitors and the selective metallization of semiconducting chips for conductivity [2]. There are several types of electroless plating such as electroless nickel (EN), electroless palladium and electroless gold. Other characteristics of EN are hardness, wear resistance, corrosion protection, and has ability to plate uniformly on complex surfaces of the substrate. This makes electroless nickel popular among other types of electroless plating and the reason why electronics industry uses this plating method for their products.

Uyemura Group is a company that produces a variety chemical plating. One of their products is Nimuden KFJ-20. KFJ-20, a very special chemical formulation which has some excellent properties as compared to previous electroless Ni-P (EN) products. KFJ-20 has two different versions which (T and S). The different between these two is the nickel thickness. Both are free from lead and toxic metals. Because of the special stabilizer present in the chemical, it makes this product gives a good deposition speed as well as internal stress. The great advantages of KFJ-20 are excellent properties of step plating and high corrosion resistance. The stability of bath is also good, which makes it one of the best Uyemura EN products.

Due to the properties of copper substrates (passive metal), it cannot catalyze hypophosphite oxidation [3]. So, there is no deposition of nickel on copper substrates. Thus, surface treatment is required to obtained nickel layer on copper substrate.

2. EXPERIMENTAL

The experiment was divided into three main stages. The first stage focused on preparation of pre-treatment solutions, nickel strike solution and KFJ-20. The pre-treatment solutions prepared were C-4000 (cleaning solution), soft etching, pre-dipping, and acid dipping. The sample (copper substrate) was cut into 5cm x 5cm sized. In the second stage, the copper substrate underwent a plating test. It started with the pre-treatments, continued with nickel strike activation for 2 min with 10 A/dm^2 and lastly, plating in KFJ-20 for 20 min with the condition of 90°C and 200 rpm of the KFJ-20 bath. The deposition speed of nickel layer was calculated and recorded. Variable parameters such as pH, temperature, plating time of KFJ-20 and current density of nickel strike were also being tested in this research. Later, in stage three, copper substrates with and without nickel strike activation were characterized to study the topography, morphology and chemical composition using FESEM and EDX.

The chemical reactions for this process are suggested such as:

$$Ni^{2+} + H_2PO_2^- + H_2O \rightarrow Ni^0 + H_2PO_3^- + 2H^+$$

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + H_2$$

Overall reaction:

$$Ni^{2+} + 2H_2PO_2^- + 2H_2O \rightarrow Ni^0 + 2H_2PO_3^- + 2H^+ + H_2$$

$$H_2PO_2^- + H^+ \rightarrow H_2O + OH^- + P$$

3. RESULTS AND DISCUSSION

3.1 Calculation of the deposition speed of nickel layer on copper substrate with and without nickel strike activation

The deposition speed of nickel layer was being calculated by using this formula**:**

$$\text{Deposition speed} = \frac{\text{Different in weight of sample (after} - \text{before)} \times 10000}{\left[\text{Area of sample (cm}^2\text{)} \times \text{Density of Ni } \left(\frac{\text{g}}{\text{cm}^3}\right) \times \text{Time of plating (min)}\right]}$$

By using this formula, the deposition speed of nickel layer for copper substrate without nickel strike activation give negative result which was -2.513 µm/h. When the nickel strike activation was implement in the step of plating, the result show positive deposition speed of nickel strike which 4.965 µm/h with semi bright appearances. This was one of the evidence that by adding nickel strike activation into normal plating process, nickel layer can be deposited on the surface of copper.

3.2 Variable parameter in KFJ-20 and nickel strike bath

3.2.1 Effect of different pH in KFJ-20 bath

Plating test with different pH value was conducted to see whether pH can affect the deposition of nickel layer on copper substrate. Figure 1 show the trend of deposition speed of nickel layer against different pH of the KFJ-20. It shows that in the condition of pH 3.5, negative deposition speed of nickel was achieved due to the potential of H_2 was more positive that nickel. In the condition of pH from 4.0 to 5.0, the deposition speed of nickel layer was increased due to the degree of nickel orientation was saturated [3,4]. In the condition of pH from 6.0 to 7.0, the deposition of nickel layer was also increased but nickel layer was flaked due to formation of $Ni(OH)_2$ on the copper substrate.

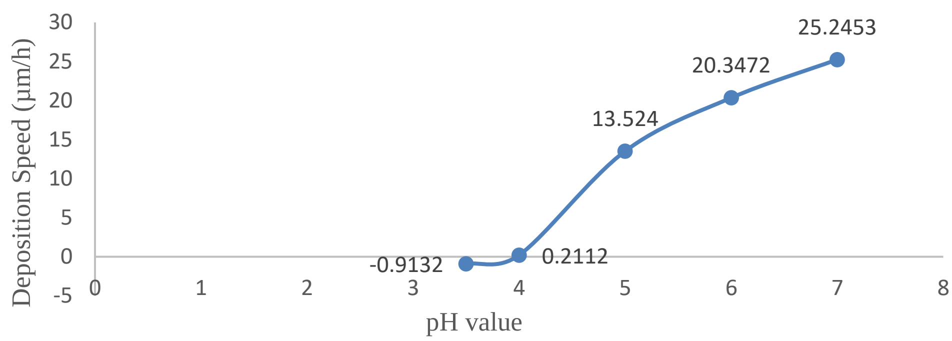

Figure 1 Deposition speed of nickel layer on copper substrate with different pH value in KFJ-20 bath.

3.2.2 Effect of temperature in KFJ-20 bath

Figure 2 show the study of temperature difference on deposition of nickel. It shows that the deposition speed of nickel layer increase from 88.0℃ to 96.0℃. This is because of the effective collision between the Ni^{2+} ion. Increasing the temperature will make the Ni^{2+} ion more rapidly collide with the surface of the copper substrate. This prove that more effective collision occurs if the temperature was increased from 88.0℃ to 96.0℃. A lot of bubble were seen as the temperature increases from 88.0℃ to 96.0℃ due to formation of hydrogen gas from the reaction.

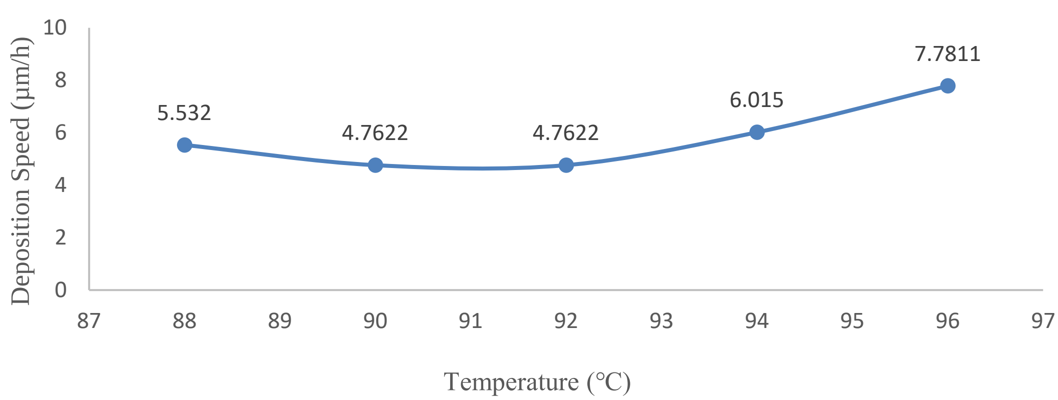

Figure 2 Deposition speed of nickel layer on copper substrate with different temperature in KFJ-20 bath.

3.2.3 Effect of plating time in KFJ-20

As can see from the formula of deposition speed, the time will not affect the deposition speed of nickel layer. This is because of the plating time in min will be converted into hour. Figure 3 show the trend of deposition speed against the plating time.

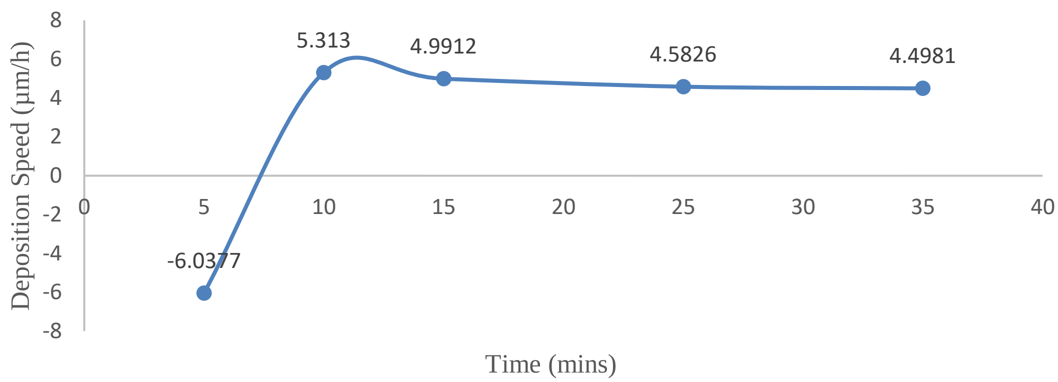

Figure 3 Deposition speed of nickel layer on copper substrate with different plating time in the KFJ-20 bath.

3.2.4 Effect of current density in nickel strike activation

As shown in Figure 4, when 2.0 A/dm^2 current density was applied to the nickel strike step, the deposition speed was negative result. As compared to 12 A/dm^2 current density, the degree of nickel orientation was more favorable. It seems that 2.0 to 8.0 A/dm^2 was too small to plate nickel film on copper substrate. This because of the negative potential in 2.0 to 8.0 A/dm^2 were higher compared to the activation overpotential. Thus, nickel film was not preferable to plate the nickel on copper substrate.

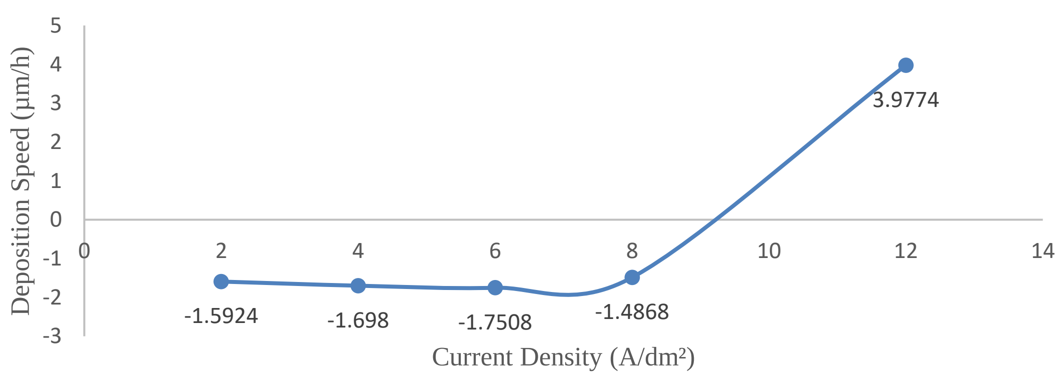

Figure 4 Deposition speed of nickel layer on copper substrate with different value of current density of the nickel strike activation.

3.3 Characterization of copper substrate with and without nickel strike activation

The morphology and chemical composition of the copper substrate without and with nickel strike activation was compared in Figure 5. As shown in Fig. 5(a) and (b), the surface appearance and uniformity of nickel layer obtained from copper substrate with nickel strike activation are comparable to those from copper without nickel strike activation. Copper with nickel strike activation have discoidal shape of particle compare to irregular shape on surface of copper without nickel activation. In addition, the chemical composition of copper substrate with and without nickel strike activation are compared in Fig. 5(c) and (d), respectively. The results show that copper substrate with nickel strike contained almost 85 wt.% of nickel compare to another one which not contained any nickel.

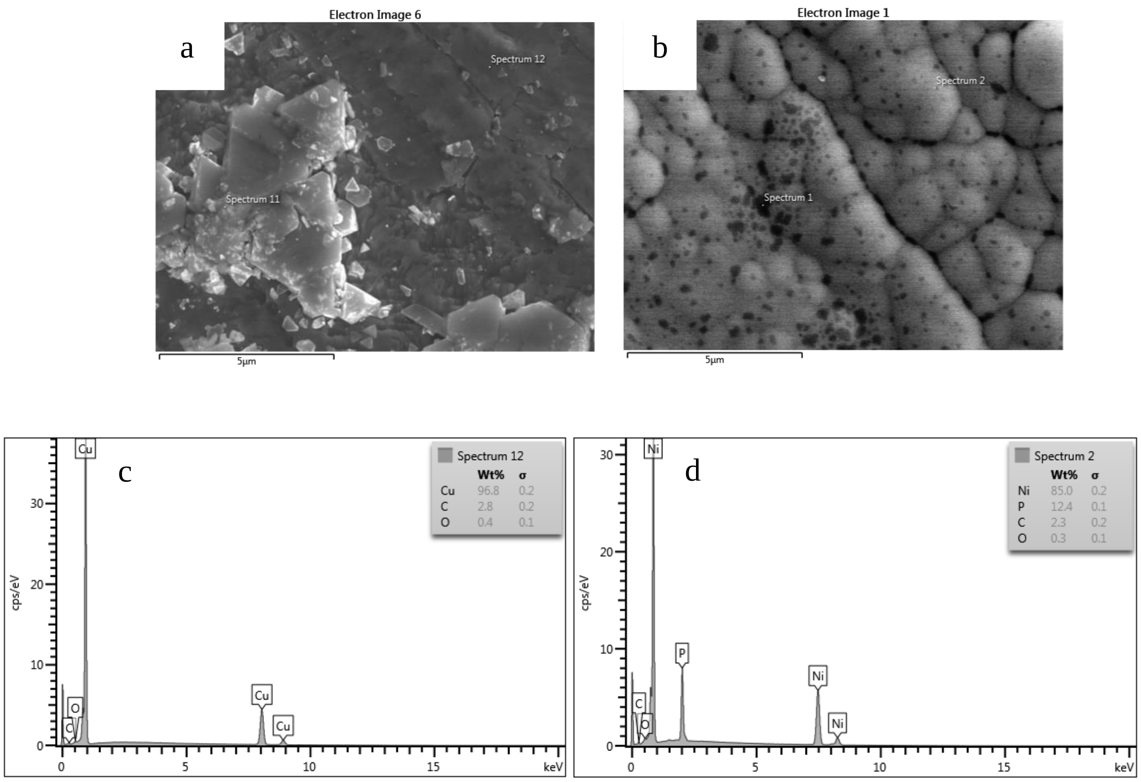

Figure 5 FESEM images (a, b) and corresponding EDX spectra (c, d) of copper substrate without nickel strike activation (a, c) and with nickel strike activation (b, d).

4. CONCLUSION

Copper substrates with and without nickel strike activation were plated with KFJ-20 bath. The nickel layer was deposited on surface of copper substrate with nickel strike activation. Various parameters were controlled such as pH condition, temperature and plating time in KFJ-20 bath to investigate whether there is a difference in deposition speed. The deposition speed was increased drastically from pH 4.0 to 5.0. The higher the temperature, the higher amount of deposition speed obtained. The longer nickel plating time, there is no trend of increasing deposition speed. When the current density was 12 A/dm^2, the deposition of nickel layer was present as compared to the other current densities. In this work, the best condition for KFJ-20 bath was pH 4.0-5.0, temperature at 90℃, plating time of 20 min, and current density of 10 A/dm^2 for the Ni strike activation and gave 85.0 wt.% of nickel.

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