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Electroless Nickel-Phosphorus Plating on Copper Substrate Using Galvanic Starter

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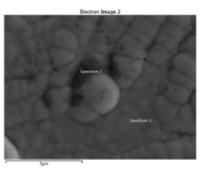
GRAPHICAL ABSTRACT



Copper substrate with galvanic starter activation

ABSTRACT

This study focusses on deposition of nickel layer onto copper substrates. Electroless nickel (EN) deposition is classified as a class of coating that is used to enhance the surface performance characteristics of a substrate. The processes include cleaning, soft etching, pre-dipping, acid dipping and electroless nickel plating. These processes are common for plating. For the first trial, there was no deposition of nickel layer onto the copper substrate with negative deposition speed, -1.7359μm/h. Galvanic starter was used for activation process, in which the iron plate was activated in H_2SO_4 bath before being touched onto copper plates. The optimum activation time for iron plate is 5 min. The time for the activated iron to touch onto the surface of copper is between 10-15 s. Hence, the nickel layer can be seen to be deposited onto the surface of copper substrate by a change of colour of the plate. The deposition speed of nickel layer is 5.3811 μm/h with semi-bright appearances. The performance of EN depends on the various parameters such as pH, operating temperature and deposition time. The standard values of those parameters are 4.4, 90ºC and 20 mins, respectively. The properties of EN plating were characterized using Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray (EDX) which show the topography and chemical composition of copper substrate. The results show that the copper substrate without iron touch (galvanic starter) has irregular shape on the surface and contain of 96.8 wt.% of copper, 2.8 wt.% of carbon and 0.4 wt.% of oxygen. For the copper substrate with iron touch, the result shows that the chemical composition is 87.3 wt.% of nickel, 10.8 wt.% of phosphorus, 1.7 wt.% of carbon and 0.2 wt.% of oxygen.

Keywords: electroless nickel plating, copper substrate, galvanic starter, H_2SO_4 bath

1. INTRODUCTION

Electroless nickel (EN) plating was developed in the early 1970's [1]. EN undergoes autocatalytic chemical reaction that deposits nickel-phosphorus coating on a metal substrate. This reaction occurs when the substrate such as copper has been stripped from its originally passive oxide layer. The most important part for EN plating is how it deposits on the surface of a plate uniformly depending on the types of substrate [2].

The process does not use external current. Sodium hypophosphite is the common reducing agent used. It can act as a better corrosion resistance. Electroless plating has several advantages than electroplating. Electroless plating has very desirable properties compared to electroplating which varies at different pH, temperature, solution composition of the bath and agitation [3]. The coating makes this process more attractive for the applications. Due to the advantages EN plating is widely used in industry and in our daily life for many types of applications such as automotive industries, aerospace and electronics (4). In addition, EN can also be used in new areas such as ceramics, fabrics, corrosion coating and catalytic surfaces.

The untreated copper substrate cannot undergo autocatalytic process. It will have low performances such as low corrosion resistance, hardness and surface structure. In order to overcome this problem, it requires a further additional method for activation of the surface during the process. The parameters such as pH and plating time of EN plating should be controlled and improved. Besides, the surface treatment also should be emphasized in order to get the better properties during the process.

2. EXPERIMENTAL

The experiment was divided into three main parts as shown in Figure 1. The first step focused on the pre-treatment of the copper surface such as soft-etching, pre-dipping and acid dipping. This was followed by the activation process before plating using galvanic starter by activating the iron plate in activation bath containing 10% of H_2SO_4. The last step was deposition of nickel from KFJ-20 onto the copper surface. Parameters such as temperature, pH and time plating was optimized. The deposition speed of nickel layer onto copper substrate will be calculated. Lastly, the copper substrate will be characterized using Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray (EDX). Figure 1 shows an overview of EN deposition process. For the overall process, the copper plate was cut before it was dipped into the C-4000T solution at 62^{0}C for 1.5 min.

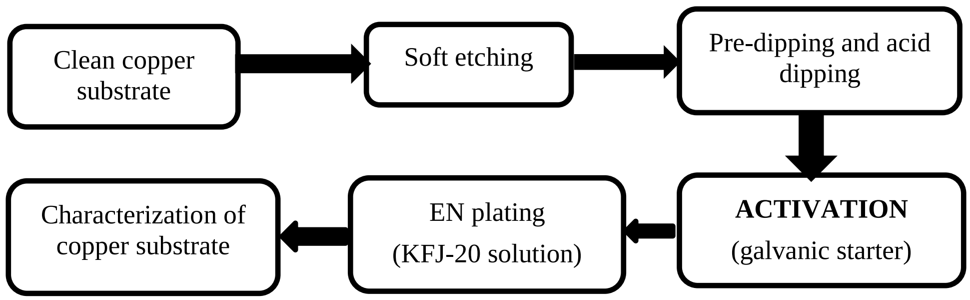


Figure 1 Overview EN deposition process.

The plate was then rinsed with deionized water three times. This is to ensure that all oxides are removed from the substrate surface. Deionized water was used in all the operations to avoid impurities and the set-up was clean. The copper plate was dried using an air blower and weighed using an analytical balance (w_1). The previous step was repeated for another 1.5 min. Then, the plate was rinsed three times before dipping it in the soft-etching for 1 min. The plate was then rinsed again and dipped in the pre-dip solution for 1 min and continuously rinsed with deionized water. This was followed by dipping in an acid dipping solution for 1 min and also rinsed with deionized water three times. Lastly, the process was continued with the iron touch (galvanic starter) activation whereby the iron plate was hanged in the H_2SO_4 solution for 5 min to activate the metal iron. Lastly, the copper plate was dipped in the KFJ-20 solution according to the plating conditions and the iron plate was touched onto the top surface of the copper plate for 10-15 s. After 20 min, the copper plate was dried and weighed again (w_2).

3. RESULTS AND DISCUSSION

3.1 Effect of Temperature

From the study conducted, temperature is one of the parameters which influences the deposition of copper substrate on the nickel plating as shown in Figure 2. Reduction and oxidation processes are occurring in an entire reaction that needs external energy for all the types of bath. Deposition on plating using hypophosphite baths was in the temperature range between 80 ºC and 96 ºC. The relationship between temperature and deposition rate of coating is independent on the acidity or alkalinity of the bath. Usually, the good deposition rate is obtained above 80 ºC. But, when the temperature is beyond 90 ºC, there is instability of the bath or decomposition may occur. Deposition is completely weak at temperature around 60 ºC but it is suitable only for metallizing non-conductors with poor resistance without melting at higher temperature. Hence, the temperature should be controlled accurate in order to get the great deposition.

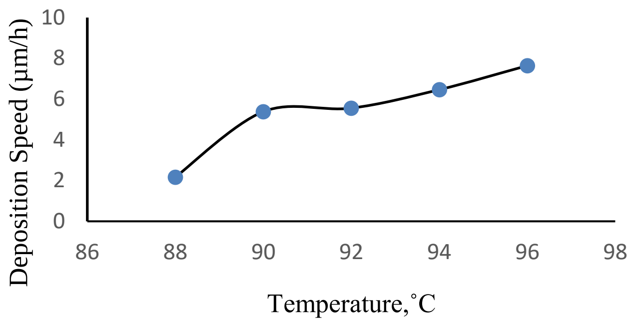


Figure 2 The deposition speed of nickel layer on copper substrate with different temperature.

3.2 Effect of pH

The effect of pH on the rate of deposition is shown in Figure 3. The decrease in pH is due to the increasing content of phosphorus in the coating. A decreasing of pH will prevent deposition and lower the power of reducing agents. At pH 3, deposition rate seems to be delayed and deposit formed around the substrate. At pH range 4 - 6.0, the deposition can be more effective. Increasing the pH may enhance the deposition rate and lower the solubility of nickel salts. So, the pH bath should be controlled at optimum value to obtain the desired deposit.

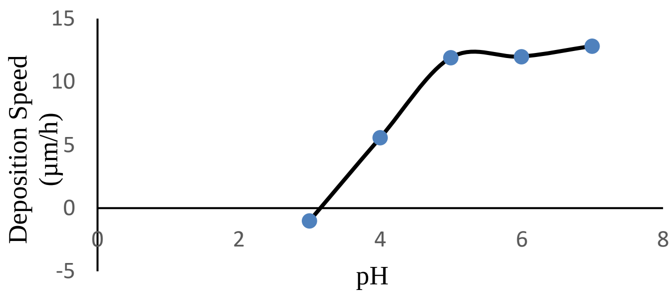


Figure 3 Deposition speed of nickel layer on copper substrate with different pH.

3.3 Effect of plating time

Plating time is the total time of substrate immersed in the bath solution. Deposition speed usually does not affect the plating time as shown in Figure 4.

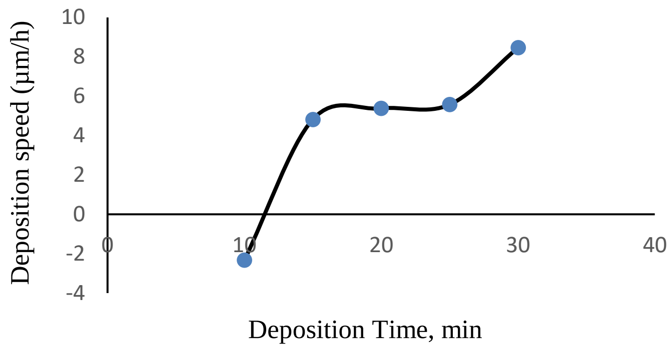


Figure 4 Deposition speed of nickel layer on copper substrate with different plating time.

3.4 Effect of Galvanic Starter

In this process, iron plate was immersed in 10% H_2So_4 solution for 5 min. This is to activate the surface of metal iron. Then, the iron plate was touched directly onto the copper substrate in the EN solution for 10-15 s. Figure 5 shows that increasing the time of iron touch, this also increase the deposition speed.

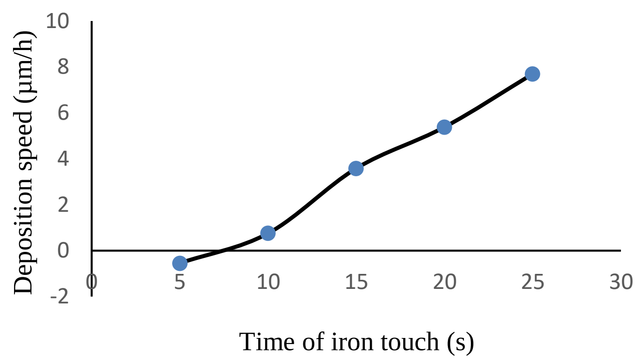
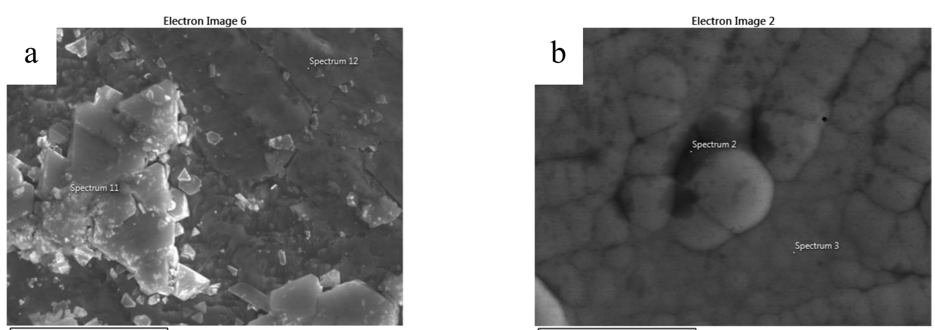


Figure 5 Deposition speed of nickel layer on copper substrate with different value of galvanic starter.

3.4 Characterization of copper substrate with and without galvanic starter activation



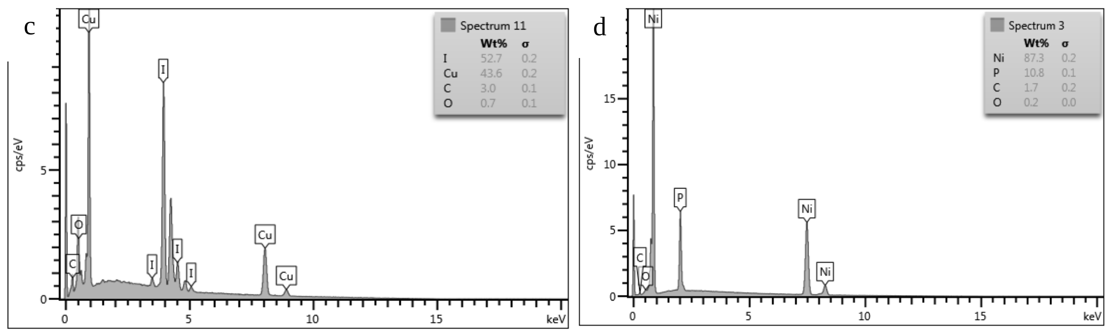


Figure 6 FESEM images (a, b) and corresponding EDX spectra (c, d) of copper substrate without (a, c) and with galvanic starter activation (b, d).

From the result, the weight percentage (wt.%) of the element contained in the copper substrate was obtained. EDX spectra in Figure 6 (c,d) show the chemical composition of copper substrates. The EDX spectrum of copper substrate without galvanic starter activation shows that the chemical composition obtained is 52.7 wt.% of iodine, 43.6 wt.% of copper, 3.0 wt.% of carbon and 0.7 wt.% of oxygen. The EDX spectrum with galvanic starter activation, the composition is 87.3 wt.% of nickel, 10.8 wt.% of phosphorus, 1.7 wt.% of carbon and 0.2 wt.% of oxygen.

4. CONCLUSION

In this study, the main objective was to determine the capability of EN on the hardness, corrosion and abrasion resistance of electroless Ni-P coating on the copper substrate. From the experiment, copper substrate with galvanic starter as activation shows positive result which is 5.3811 µm/h, but the copper substrate without galvanic starter shows negative deposition speed which is -1.7359µm/h. It shows that deposition speed give good performance when the galvanic starter activation was used. The parameters such as pH, temperature, plating time and galvanic start are the main parameters in enhancing the deposition rate on the surface. For the temperature, the higher the temperature, the higher the deposition rate of the substrate. As a conclusion, the standard condition for EN plating are pH 4.4, temperature 90ºC, plating time 20 min and activation time for galvanic starter 10-15 s.

REFERENCES

[1] Latt, K. M. (2003). *Effects of surfactants on Characteristics and Applications of Electroless Nickel-Phosphorous Deposits*: 1-98.
[2] Aaron Edward Rain, *Determining Electroless Nickel Depostition Rates In-situ*, (2012): 1-59.
[3] Sudagar J., J. Lian, and W. Sha (2013), Electroless nickel, alloy, composite and nano coatings – A critical review. *Journal of Alloys andCompounds*: 185-204, Elsevier.
[4] J. N. Balaraju, T.S.N. Sankara Narayanan And S.K. Seshadr, (2003), Electroless Ni–P composite coatings, *Journal of Applied Electrochemistry 33*: 807-817, Kluwer Academic Publishers.
[5] Sanjib Kundua, Suman Kalyan Dasa and Prasanta Sahooa, (2014), Properties of electroless nickel at elevated temperature-a review, Science Direct, : 1698-1974, Elsivier.
[6] N. M. Martyak and J. Lou, (2001), Limitations Of Nickel Hypophosphite Electroless Nickel Processes,ATOFINA Chemicals, Inc, *Trans IChemE*, Vol 79: 278-282.
[7] Dr Ing. N. V. Mandich, CEF, AESF Fellow, *Surface Preparation of Metals Prior to Plating:* 1-68, 2002.
[8] G. O. Mallory, (1990), The Fundamental Aspects of Electroless Nickel Plating, *Chemistry Engineering*: 1-56, Chapter 1.