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The effect of current density on mechanical properties of electroplated thin copper foil

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Abstract

Thin copper foils are commonly used in arts, crafts, and manufacturing industries. Although copper electroplating processes have been widely studied, most focused on either copper in bulk material form factor or copper in extremely thin film shape. Thin copper foils are considered neither bulk material nor thin film; thus, it is estimated that they will have unique properties. This study aims to investigate the mechanical properties of thin copper foils coated by electroplating processes. The investigations were conducted experimentally by performing electroplating at current densities of 2, 3, and 4 A/dm^2. The copper foil specimen having a dimension of 10 mm wide, 0.2 mm thick, and 125 mm long were electroplated in a bath of copper sulfate, sulfate acid, chloride acid, and copper brightener mixtures for 60 minutes powered by a 30 A DC power supply. The hardness and tensile test diagrams were studied. The strain, yield stress, and ultimate tensile stress data extracted from the tensile test diagram were compared with other previous studies found in the literature and fitted with linear regression lines. The relationship of those parameters with current density has been successfully obtained. The hardness, strain, and yield stress of the electroplated copper foil increased with larger current densities used in the electroplating process except for the ultimate tensile stress, which was found to be slightly decreased with current densities. The optimum operating condition for obtaining the best results was found at a current density of 3 A/dm^2.

Keywords:
thin copper foil, current density, hardness test, tensile test, yield stress

1 Introduction

Thin copper foils have long been used not only in the arts and crafts industry but also in electrical and manufacturing applications. The manufacturing of transformers, electrical motors, and generators relies heavily on the thin copper foils as the main raw material for making the core part of the equipment. The battery industry also uses thin copper foil to manufacture various Li-ion batteries often found in many electronic appliances and electric vehicles. Hence, it is interesting to investigate its mechanical properties and how to improve them.

Many ways can be done to alter the mechanical properties of the thin copper foils. One of them is by coating the foil using electroplating processes. Electroplating on many different types of material in various shapes has been widely studied in the past. Extending the tool life of a tungsten carbide micro-end-mill tool by depositing nano-sized SiC particles using electroplating was studied by Park et al. [1]. They incorporated organic additives saccharin and ammonium chloride to enhance the matrix density of the electroplating bath in improving the smoothness of the electroplated surface. They suggested that electroplating Ni/SiC coating on the micro-end-mill tool would extend its life by at least 25%. However, little information was provided about the properties or microstructure of the electroplated surface of the tool. Read et al. [2] presented detailed morphology, microstructure, and mechanical properties of electrodeposited copper in their paper. They utilized X-ray diffraction, electron backscattered diffraction, imaging in a field emission scanning electron microscope (FESEM), and micro tensile testing. They succeeded in revealing the detailed microstructure of the electrodeposited surface up to grain and subgrain levels. Their study was also supported by the stress-strain curve that explains their study quantitatively. The mechanical and electrical properties of the electroplated copper used for MR-imaging coils were studied by Uelzen et al. [3]. They created an electroplated copper film on a catheter using a sulfate electrolyte composed of sulphuric acid (H2SO4) and copper sulfate (CuSO4⊙ 5H2O). They investigated the influence of current density and temperature in the electroplating process on the deposition rate, electrical conductivity, hardness, and tensile strength of the electroplated medical devices. They recommended that a current density of 6 mA/cm^2 and a temperature of 40 °C would be best for fabricating the micro coil and catheter tip.

Xiang et al. [4] suggested that the properties of thin material are different from those of bulk material, even from the same type of material. Hence, careful attention and treatment must be given when performing electroplating on the material of such shape. Luo et al. [5] studied thin film of electroplated nickel (Ni) used for microelectromechanical applications. They measured Young's modulus for such material based on various temperatures and current density. They found that ammonium and sulfate ions by hydrolysis that was used in their experiments were the cause of the drop of Young's modulus from 205 GPa at a temperature less than 60 °C to 100 GPa at 80 °C and 205 GPa at a current density of 2 mA/cm^2 to 85 GPa at 30 mA/cm^2. Similar work on measuring the mechanical properties of electroplated thin material was done by Baek et al. [6]. They investigated Young's modulus, residual stress, and stress gradient of electroplated gold thin films using surface micromachined beam structures. They used UV-LIGA surface micromachining and dry-release methods to create cantilever and bridge beam structures of various lengths. They found that Young's modulus of the thin film was smaller than the bulk Young's modulus, and this discrepancy changed slightly depending on the deposition current density. They recommended that this phenomenon be considered when fabricating micro electro-mechanical systems (MEMS) devices.

The current density has been a key factor in the electroplating process, which has been thoroughly studied, tested, and proved by many literatures [7]–[10]. Effendi [7] investigated the impact of current density on the thickness of zinc electroplated on low-carbon steel. He used cylindrical shape low carbon material having a diameter of 12 mm and a length of 40 mm with a total of 20 samples tested. He used an electrolyte mixture of zinc oxide, KCN, and sodium hydroxide with a total concentration of 75 grams/liter. He tested various current density settings ranging from 1 to 9 A/dm^2 while maintaining the electroplating time fixed at 15 minutes for all tests. He found that the amount of the zinc material electroplated on the surface of the base material strongly depends on the current density used in his experiments. He suggested a linear relationship between current density and the thickness of the electroplated coating. Supriadi et al. [8] did a similar work to Effendi's paper. They investigated the influence of current density and electrolyte temperature on a similar base material, medium carbon steel, although the electroplating material was different. They used pure copper as the coating material with sulfuric acid electrolyte. They varied the current density from 2 to 5 A/dm^2 while the electrolyte temperature was tested at 30, 40, and 50 °C. Compared to Supriadi work, their electroplating time was kept constant at a shorter duration of 10 minutes. They found that the optimum operating condition for conducting their electroplating experiments was at a current density of 3 A/dm^2 with an electrolyte temperature of 40 ºC. Yamamoto et al. [10] studied the electroplated nickel used for manufacturing MEMS components. They investigated the effect of current density on the morphology, average grain size, Vickers hardness, micro-mechanical property, and thickness distribution. They used pillar-type specimens with a height of 20 µm and a square cross-section of 10×10 µm. They found a minimum value of grain size of 349.8 nm when the current density was increased from 10 to 20 mA/cm^2. They observed that the electroplated nickel at 20 mA/cm^2 showed a high growth rate of 0.296 µm/min and the highest Vickers hardness at 371 HV with yield stress obtained from a compression test of 1.3 GPa. Sudarsono et al. [9] conducted similar work to Effendi [7] and Supriadi et al. [8]. They also used low-carbon steel as the base material, but they tested several combinations of electroplated material such as copper, nickel, or a mixture of copper-nickel. They investigated the effect of current density, but only the hardness of the resulting electroplated material was tested, while other mechanical properties were not covered in their paper. They obtained the highest hardness at 290 kg/mm^2 on a nickel coating specimen with a current density used of 5 A while the lowest hardness was observed at 111 kg/mm^2 on a copper coating specimen with a current density of 12 A.

Other works that need to be cited and mentioned that are strongly related to the research in this article are the ones that used copper thin film as the base material and also copper as the coating material, although the method used and the parameters investigated are different [4], [11]–[14]. Volinsky et al. [11] tested the microstructure and mechanical properties of electroplated copper thin films having various thicknesses ranging from 0.2 to 2 microns. Their microstructure images were obtained using Atomic Force Microscopy and Focused Ion Beam Microscopy. They measured the elastic modulus and hardness of their electroplated material using the continuous stiffness option (CSM) of the Nanoindenter XP and found an elastic modulus of 110 to 130 GPa and hardness of 1 to 1.6 GPa. Xiang et al. [4] studied the mechanical properties of free standing electroplated copper thin films having thickness varies from 0.9–3.0 µm. They examined the detailed microstructure of the electroplated film using Focused Ion Beam microscopy (FIB) and found a slight increase in the stiffness of the copper film due to alterations in the crystallographic texture and the elastic anisotropy of copper. Miura et al. [12] studied the fluctuation in mechanical properties of electroplated copper thin films used for three-dimensional electronic modules. They compared the copper thin films formed by cold rolling with those created by electroplating using tensile test and nano-indentation. Their microstructure images were obtained by using a scanning electron micrograph. They found that both Young's modulus and tensile strength of the films had drastic variations depending on the microstructure in the electroplated films. Jeong et al. [13] investigated the mechanical properties of intermetallic compounds formed at the interface between a tin bump and an electroplated copper thin film. They used $CuSO_4$ electrolyte for the copper film and $Sn(RSO_3)_2$ electrolyte for the tin film while the same current density of 20 mA/cm^2. They measured Young's modulus using nanoindenter DCM SA-2 made by MTS Corp. They found that the greater thickness ratio of the IMC layer may alter the dominant fracture mode from the fatigue crack. More extensive fatigue tests were conducted by Watanabe et al. [14]. Using the Manson-Coffin law, they projected the thermal fatigue life of the electroplated copper thin film for the through hole on a printed circuit board. They discovered that the fractures caused by thermal fatigue were intergranular fractures at random grain boundaries. The fatigue life of electroplated copper film was reduced by about a third when the temperature increased from 298 K to 398 K.

In this article, instead of using thin copper film, thicker materials of thin copper foils were used in the electroplating process using copper as coating material as well. The character of the resulting product from the electroplating is expected to be somewhat between the bulk material and the thin film mechanical properties. The method applied in this study is a more traditional way with tensile and hardness tests, which will be further explained in subsequent sections.

2 Research methods

The electroplating process for manufacturing the samples tested in this study was conducted according to what Van Vlack [15] suggested, as illustrated in Fig 1. The copper foils to be electroplated were placed as the cathode which were then immersed in a bath consisting of copper sulfate 45 ml/l, sulfate acid 45 ml/l, chloride acid 0.1 ml/l with copper brightener type A 0.8 ml/l and type B 2 ml/l while the anode used was made of copper. This electroplating system was powered by a 30 A DC power supply. The copper foils used in the experiments were 10 mm wide and 0.2 mm thick, which were cut into several pieces of 125 mm long.

The chemical reaction of the copper deposition on the specimen at the cathode part of the electroplating system with the bath of electrolyte mixture used in this study can be written as Eq. (1) and while the oxygen gas formation at the anode part can be written as Eq. (2).

$$Cu^{2+} + 2e \rightarrow Cu \tag{1}$$

$$H_2O \rightarrow 2H^{+} + O_2 + 2e \tag{2}$$

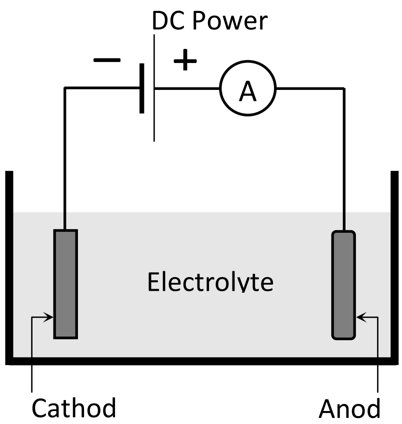

Fig 1. Illustration of the electroplating process in this study

Before starting the electroplating process, the specimens were cleaned with distilled water to remove any impurities. The electroplating process was conducted for 60 minutes at a constant room temperature of 25 ºC with three different current density settings, 2, 3, and 4 A/dm^2. The current density used in this study is defined as Eq. (3).

$$i = \frac{I}{A} \tag{3}$$

where I is the current applied in ampere (A) and A is the surface area of the specimen in square decimetres (dm^2). After the electroplating had been completed, the specimens were washed, rinsed, and dried before examining their mechanical properties.

The hardness of the electroplated specimens was measured by micro indentation test using Micro Vickers hardness test equipment FUTURE-TECH FM-800. The illustration of the measurement system is shown in Fig 2. Vickers hardness test equipment uses a diamond pyramid-shaped indenter that will make a quadratic footprint when gradually pressed on the surface of the specimens. Vickers hardness number, Hv, for the specimens was calculated using the relationship that was used by Uelzen et al. [3] for similar specimens,

$$HVN = \frac{F}{26.43h^2} \quad (4)$$

The load used in this study was F = 200 gf with a loading time of 10 s. To ensure consistency and to avoid part of the specimens that might inconsistently be electroplated, the hardness tests were repeated three times for each type of sample. The final values taken were the averaged value from three hardness test readings. The standard deviation of the reading of the three hardness tests would be plotted as an error bar for each type of sample.

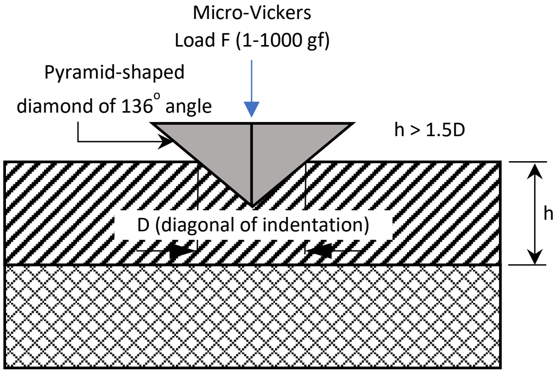

Fig 2. Illustration of Micro-Vickers hardness test

The strength of the copper electroplated results was determined by applying tensile tests using Universal Testing Machine HUNGTA, HT8503. Due to the thin shape of the specimens, special care was given to avoid incorrect readings, and the tensile test was performed in a slightly different way according to the tensile testing standard specifically for thin specimens, ASTM D3039. The example of the electroplated specimens before and after the tensile test are shown in Fig 3. The tensile tests will produce stress-strain graphs that are useful not only in determining the modulus and the strength of the electroplated results but also in observing whether the specimens are ductile or brittle.

(a)

(b)

Fig 3. The electroplated specimen (a) before and (b) after the tensile test.

3 Results and discussion.

The effect of increasing current density on the hardness of electroplated copper foils is plotted in Fig 4. As seen in the figure, the hardest specimen was the one deposited using a current density of 3 A/dm^2. The overall effect of increasing the current density appears to be slightly increasing the hardness of the electroplated specimens. As can be seen from the error bar that shows the standard deviation of the repeated results, uncertainty and variation were relatively low and acceptable. Compared to previous works found in the literature, there have been no agreement and general consensus on how the current density affects the hardness of electroplated material, as some studies found the hardness decreases while other works found it increases. It all depends on the type of material to be deposited, as in some cases, the coating materials are harder while others are softer than the base material.

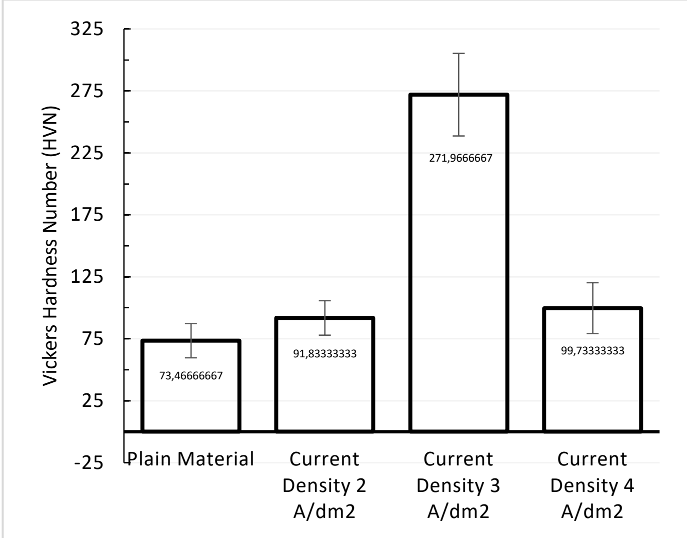

Fig 4. Hardness test results from electroplated copper foil specimens at various current densities.

Rasyad and Arto [16], who studied chrome electroplated on low carbon steel, suggested that the hardness might increase, although their charts are quite difficult to interpret as the hardness was not directly plotted against current density. A more straightforward conclusion was given by Sudarsono et al. [9], who investigated copper-nickel electroplated on low carbon steel. They also found that the hardness increases against current density. According to Uelzen et al. [3] who studied copper electroplated on MR imaging coils, the increase of hardness against current density was due to defects in the crystal structure of the electroplated material. On the contrary, Yamamoto et al. [10], who investigated nickel electroplated on MEMS components, found that the decrease of hardness against current density was due to a decrease in grain size and grain coarsening that leads to softening of the electroplated specimens.

The curve-fitting plot of the hardness of the electroplated copper foil, as shown in Fig 5, confirms that the current density increases the hardness, although it is not very significant. By applying a linear regression approach to the data, the relationship between the hardness, HVN, and the current density, I, can be expressed into equation as Eq. (5).

$$HVN = 21.743i + 85.329 \quad (5)$$

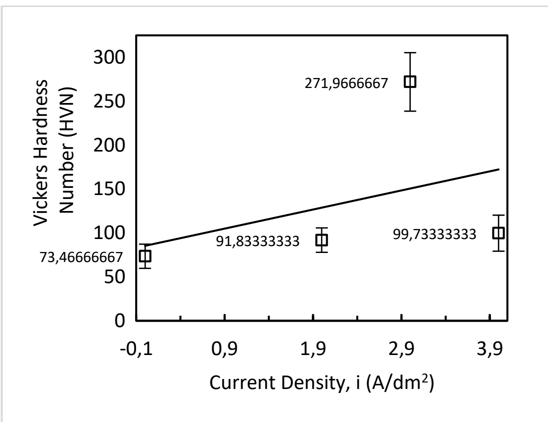

Fig 5. The curve fitting of the hardness of electroplated copper foils.

The tensile test results of the specimen with and without the electroplating process are plotted in Fig 6. As seen from the figure, the plain copper foil shows a common brittle-type pattern of typical tensile test results with little deformation and strain of less than 1% before necking and fracture occur (blue line). All part of the plain material line appears to be elastic deformation as it is linear with no clear region of plastic deformation. Hence, the yield point of this specimen is not visible, and it was determined by using the offset method. The necking is also not visible, and it appears the fracture of the specimen occurs at the maximum stress. On the contrary, all electroplated copper foil specimens show a ductile-type pattern of tensile test results, with specimen electroplated at a current density of 3 A/dm^2 show the largest ultimate tensile stress. Unlike plain copper foil, all electroplated specimens show clear linear and nonlinear parts of the lines. Thus, the yield points were taken as the boundary when the lines started to become nonlinear. However, the necking part of the lines is subtle, and fractures of the electroplated specimens occur just shortly after the maximum stress points.

Inconsistency of the tensile test results of electroplated copper is also observed in the literature. Miura et al. [12] compared electroplated with cold rolled copper thin films of 10 μm thick. They observed the super plasticity phenomenon in the tensile test results of the electroplated copper thin film with an extremely long nonlinear line of plastics region. They argued that the strain hardening usually found in bulk material was absent due to cooperative grain boundary sliding. They observed that strain hardening occurred in the cold rolled copper thin film samples, and it demonstrated the tensile test behavior of bulk copper, which is similar to the results in this study. Kim et al. [17] also compared electroplated with annealed copper films of 10 μm. Their tensile test results for the electroplated copper film were similar to the results in this study, and they did not demonstrate super plasticity behavior. They found that the maximum stress of the electroplated samples was higher than those of annealed samples due to the smaller grain size.

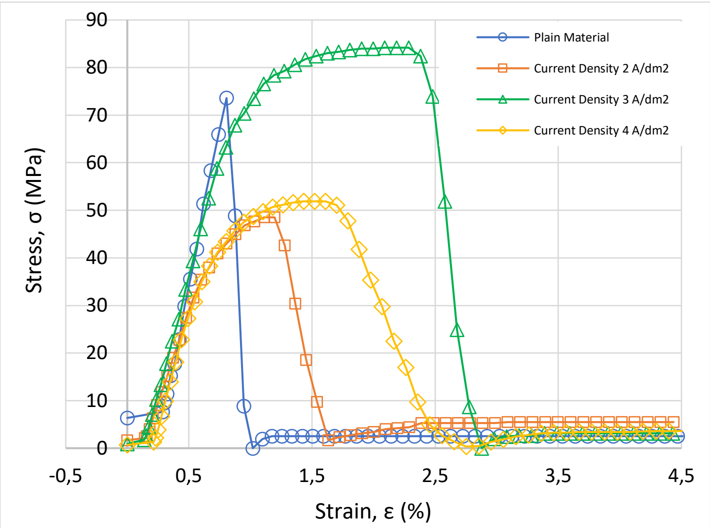

Fig 6. The stress-strain diagram obtained from the tensile tests of electroplated copper foils.

The length of the specimens before and after the tensile tests were measured to obtain the data for calculation of the maximum strains plotted in Fig 7. The overall trend of the data in the figure shows that the strain increases against the current density applied with the electroplated copper foil at a current density of 3 A/dm^2 having the largest strain. The curve fitting of the data can be achieved by a linear regression line with relatively good accuracy, $R^2 = 0.8647$. The relationship between the strain, ε as a function of current density, I, can be described by the equation of the linear regression line as Eq.(6).

$$\varepsilon = 0.5512i + 0.8839 \tag{6}$$

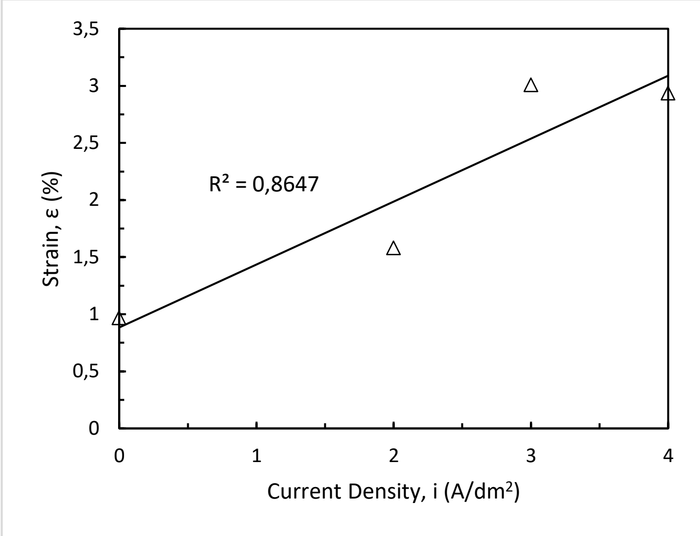

Fig 7. The strain of the electroplated copper foil at various current density

The yield points and the maximum points in the tensile test diagram shown in Fig 6 are extracted to obtain the yield stress, σ_{yield}, and the ultimate tensile stress, σ_{uts} which are then plotted together in Fig 8. It is obvious that the ultimate tensile stresses are higher than yield stresses, as the previous one occurs before the last one. However, a close look at both curves reveals an interesting feature: the gap between both stresses appears to get narrow with increasing current density of the electroplating process. Both yield stress and ultimate tensile stress reach a maximum at a current density of 3 A/dm^2, although the trend of both stresses is different. In order to get a more accurate interpretation of the trend of both data, curve-fitting processes are performed and the results are plotted in Fig 9 and 10.

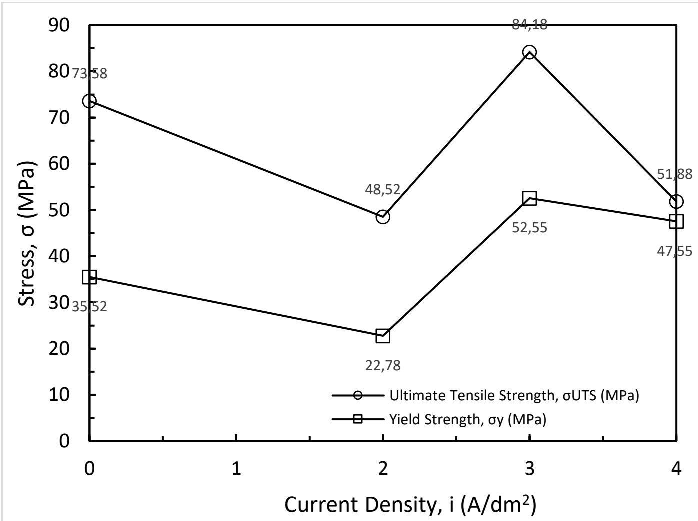

Fig 8. The yield and ultimate tensile strength at various current density

The curve fitting line of the yield stress data, as plotted in Fig 9, clearly shows that current density increases the yield stress of the electroplated copper foils and the steep gradient of the fitting line shows that the effect is quite significant. A simple linear regression approach shows good agreement between the fitting line and the experimental data of the yield stress with an accuracy of $R^2 = 0.8655$. The relationship between current density, I, and yield stress, σ_{yield} can be obtained from the equation of the linear regression line as Eq. (7).

$$\sigma_{yield} = 10.742i + 10.281 \tag{7}$$

Unlike yield stress, when the ultimate tensile stress data are fitted with a linear regression line, the overall trend shows a decreasing pattern with a negative gradient value (Fig 10).

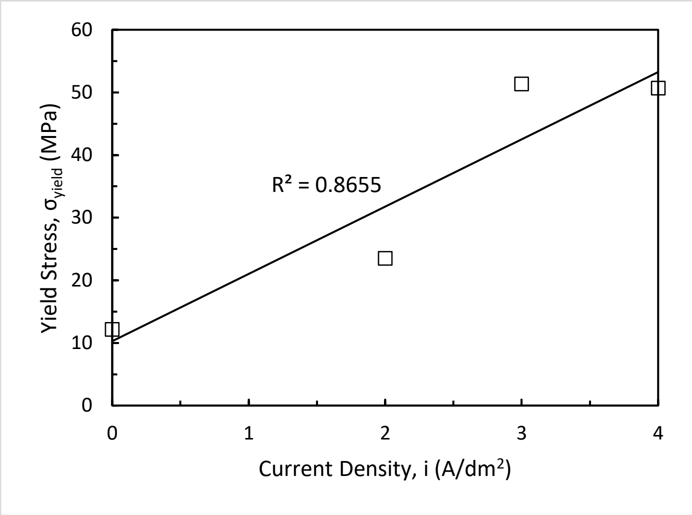

Fig 9. The curve fitting of the yield stress of electroplated copper foils.

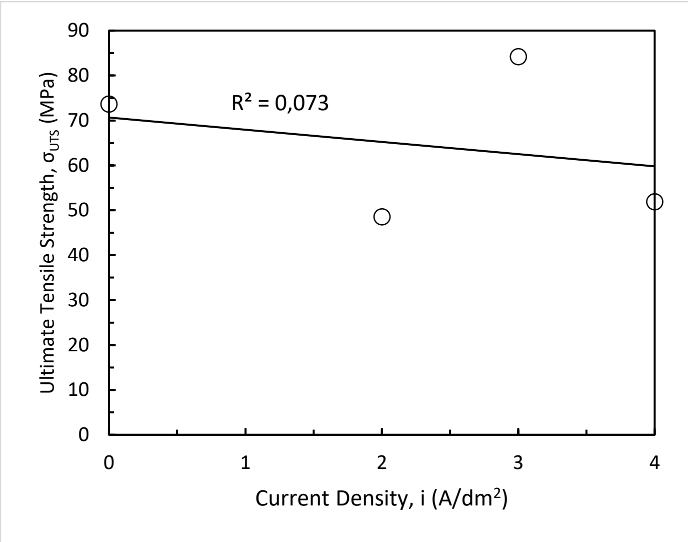

Fig 10. The curve fitting of the ultimate tensile strength of electroplated copper foils.

However, the effect of current density is not too significant as the fitting line appears only slightly decreasing. Although the accuracy of the fitting line is not relatively high, the relationship between current density, I, and ultimate tensile stress, σ_{uts}, can still be derived from the linear regression line as Eq. (8).

$$\sigma_{uts} = -2.7154i + 70.65 \tag{8}$$

Unlike the hardness and tensile tests diagram, the yield and the ultimate tensile stress results in the literature are quite consistent, although disagreement can still be found. Most other studies in the literature plot elastic modulus or young's modulus data instead of yield stress data. The Young's modulus applies along the elastic part of the tensile test curve, whereas the yield stress lies on the boundary between the elastic and plastic parts. Thus, it is safe to assume that yield stress is comparable to young's modulus. Uelzen et al. [3], who studied copper electroplated on MR imaging, suggested that Young's modulus increases against current density, and their plot are quite similar to the yield stress plot in Figure 9. Their plot of ultimate tensile stress also shows a slightly decreasing trend, which is very similar to the trend in Fig 10. On the contrary, Luo et al. [5], who investigated nickel electroplated for MEMS applications, show that the Young's modulus decreases with current density, which is the opposite of the trend in Figure 9. Rasyad and Arto [16], who studied chrome electroplated on low carbon steel, also presented stress data, although it is unclear whether they are yield stress data or ultimate tensile stress data. Their stress data for chrome electroplated specimens show a decreasing pattern similar to the trend of the ultimate tensile stress plot in Fig 10.

Other important points that may have affected the results obtained in this study are variables that were kept constant such as the electrolyte temperature and the electroplating time. These variables are directly related to the quality as well as the quantity or the thickness of the electroplated material deposited on the surface of the copper foil. The distance between anode and cathode during electroplating process may also affect the results and there must be an optimum distance that can produce the best results. This would be interesting to investigate in the future.

4 Conclusions

Current density, as one of the key parameters in the electroplating process, has been tested and proved to have an important effect on the mechanical properties of electroplated copper foil. The hardness of the copper foils increases when the current density is increased from 2 to 4 A/dm^2, with the maximum hardness obtained for electroplated samples at a current density of 3 A/dm^2. The tensile test stress-strain diagram for electroplated specimens showed a ductile-type pattern with clear elastic, plastic, necking, and fracture parts, while the plain copper foil showed a brittle-type pattern with less obvious yield point, necking, and fracture parts. The strain, yield stress, and ultimate tensile stress data were extracted from the diagram, and the relationships between current density applied in electroplated copper foil, hardness, strain, yield stress, and ultimate tensile stress were successfully obtained by applying curve fitting using linear regression equations. The overall effect of increasing current density was an increase in strain and yield stress and a decrease in ultimate tensile stress with the optimum value of 3 A/dm^2 for obtaining the best electroplating results.

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