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A comparison between destructive and non- destructive techniques in determining coating thickness

F I Haider, Suryanto, M H Ani and M H Mahmood

Department of Manufacturing and Materials Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia

Email: surya@iium.edu.my

Abstract. Measuring coating thickness is an important part in research works related to coating applications. In general, techniques for measuring coating thickness may be divided into destructive and non-destructive methods which are commonly used depending on the applications. The objective of this study is to compare two methods measuring the coating thickness of electroplating copper coating on the austenitic stainless-steel substrate. The electroplating was carried out in a solution containing 200 g/L $CuSO_4$, 100 g/L H_2SO_4 at room temperature and current of 40mA/cm^2 during 20,40,60,80 and 100 mins as coating periods. And the coating thickness was measured by two methods, cross sectional analysis as a destructive technique and weight gain as a non-destructive technique. The results show that at 20 mins coating time interval, the thickness measured by cross sectional method was 16.67 µm and by weight gain method was 17.37 µm, with difference of 0.7 µm and percentage error of 4.11%. This error increased to 5.27% at 100mins time interval, where the values of the thickness measured by cross sectional and weight gain were 86.33 µm and 81.9 µm respectively, and the difference was 4.43 µm. Moreover, though the weight gain method is fast and gives the indication for the termination of a coating process, information regarding the uniformity, porosity and the presence of cracks cannot be obtained. On the other hand, determining the coating thickness using destructive method will damage the sample.

1. Introduction
Coatings applied on a material substrate provides properties not inherent in the material that include corrosion and wear resistance, conductivity, color, and solderability. Also, the final product’s cost depends not only on the appropriate selection of coating material, but also on the coating amount applied and the final surface texture. In this sense, the amount of coating applied is the coating thickness which is essential to the product’s final use and price. Measuring the coating thickness is a significant part in researches involving coating applications. Generally, techniques for measuring coating thickness may be divided into non-destructive and destructive techniques. For the non-destructive case, there are five commonly described methods namely; X-ray fluorescence, beta backscatter, eddy-current, magnetic induction and weight gain which arranged from highest to lowest cost respectively.

First of the non-destructive techniques is the X-ray fluorescence, which is considered as the most precise measurement method. It is used to measure the thickness of small-diameter parts or dual coatings such as gold and nickel over copper. When a material is subjected to x-ray bombardment, some of its electrons will gain energy and leave the atom, releasing a photon of x-ray energy known as x-ray fluorescence. The energy level or wavelength of fluorescent x-rays is proportional to the atomic number of the molecule and is characteristic for a peculiar fabric. The quantity of energy released will be dependent upon the thickness of the material being measured [1-2].

The second method is beta backscatter that measures many typical thickness applications, including gold on nickel, copper on epoxy, silver on copper, titanium nitride on steel, and tin-lead alloys. This method is similar to the X-ray fluorescent in that the area tested is the target of radiation, and the energy emitted from the surface is measured. Beta rays are electrons emitted from unstable radioisotopes. The electrons penetrate the plating material and reflected back (back scatter) toward the source. They are collected and counted with a Geiger-Mueller tube for conversion to coating thickness [3].

The third method is eddy current which is used to measure the thickness of nonconductive coatings on non-ferrous metal substrates. Eddy current inspection is based upon the principles of electromagnetic induction, and in turn has many similarities to the electromagnetic induction test method. Magnetically induced eddy currents generate an opposing magnetic field, which changes the circuit impedance and the output voltage. The change in output voltage is applied to calculate the coating thickness [4-5].

Fourth is magnetic induction, which is employed to quantify the thickness of a non-magnetic coating (zinc, cadmium, paint, powder coating, etc.) over a steel substrate. The execution of this test method is straightforward; a measurement probe is placed directly on the surface of the test specimen. Upon placement of the probe, the linear distance between the contact point of' the probe (with the coating surface) and the substrate is then measured [6].

The last non-destructive method is the weight gain, which is used only if the coating distribution is uniform. The thickness can be determined by weight of the sample before and after coating to measure the mass of coating and calculate the surface area of the sample and use the following equation [7].

$$Thickness\ (\mu m) = \frac{M\ (g) * 10^4}{A\ (cm^2) *\ \rho\ (\frac{g}{cm^3})} \qquad Eq\ (1)$$

Where M is the mass of coating, A is the surface area and ρ is the density of the coating material.

On the other hand, the most commonly used destructive method to measure the coating thickness is the cross-sectioning based method. In this case, coating thickness can be determined by cutting the coated sample and then view the cut microscopically by optical microscopy or scanning electron microscopy. It can also be measured by making a geometrically designed incision through the coating and viewing its cross-section with a scaled magnifier. A special cutting tool is used to make a small precise V-groove through the coating and into the substrate. This destructive method allows direct observation and is often the only means to identify multiple coatings economically, provided that alternating colors have been used. Gauges are available that come complete with cutting tips and illuminated scaled magnifier. ASTM D4 I38 outlines a standard method of this measurement system [7].

In general, a non-destructive technique is a process of inspecting, testing, or evaluating the coating, components or assemblies for discontinuities, or differences in characteristics without destroying the serviceability of the part. In other words, when the inspection or test is completed the part can still be used. In contrast to non-destructive, other tests are destructive in nature and are therefore done on a limited number of samples, rather than on the materials, components or assemblies being put into service.

This paper presents a comparison study between weight gain method as non- destructive technique and cross sectional based method as destructive technique in determining coating thickness of copper electroplating.

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2. Experimental procedures

Austenitic stainless-steel grade 316L and 99.91 % pure copper samples with dimensions of 20 mm x 20 mm x 2 mm, were used as cathode and the anode respectively. The electroplating was carried out in a solution containing 200 g/L $CuSO_4$, 100 g/L H_2SO_4 at room temperature and current density of 40 mA/cm^2 during experimental processing times of 20, 40, 60, 80 and 100 mins. All the stainless-steel samples were polished up to 1000 SiC paper, and ultrasonically cleaned in acetone for 5 mins before being coated [8-9]

Coating thickness was determined using two techniques, weight gain as non-destructive and cross sectional based as destructive method. For weight gain, the sample was weighed by using 4-digit electric balance before and after coating to calculate the mass of coating with the equation:

$$M(g) = W_2 - W_1 \qquad \text{Eq (2)}$$

Where M is the mass of coating, W_2 and W_1 are the weights of the sample after and before coating respectively. The coating thickness was measured by the equation:

$$Thickness\ (\mu m) = \frac{M\ (g) * 10^4}{A\ (cm^2) *\ \rho\ (\frac{g}{cm^3})} \qquad \text{Eq (3)}$$

Where the area of each sample was (9.6 cm^2), and the density as $\rho = 8.96\ g/cm^3$. All measured thicknesses are listed in table 1.

In the cross-sectional method, coating thickness was determined by cutting the coated sample using the EDM wire cutting machine and viewing the cut microscopically by optical microscopy which is equipped with scale magnifier. The cross-sectional image is shown in figure 1. Measurements were taken in 10 locations across the cross-section of the sample on every side for every sample, and the average value was calculated. All thicknesses measured are listed in table 1.

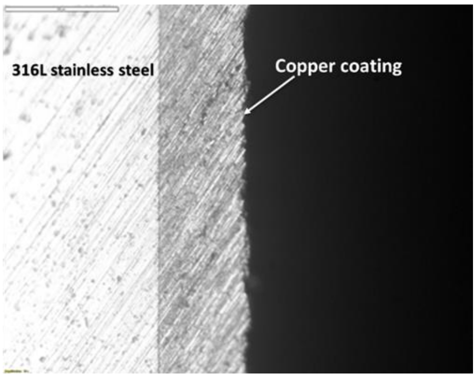

Figure 1. Optical microscopy image of cross-section copper coating on 316L substrate.

Table 1. Thickness measured by weight gain and cross-sectional methods.

Coating Time (mins)	Thickness by weight gain (μm)	Thickness by Cross sectional (μm)	Difference (μm)	Average Thickness (μm)	Error in %
20	17.37	16.67	0.7	17.02	4.11
40	33.77	35.24	1.47	34.505	4.26
60	51.42	54.06	2.64	52.74	5.01
80	69.5	66.09	3.41	67.795	5.03
100	81.9	86.33	4.43	84.115	5.27

The percentage error between the thicknesses in the two methods was calculated by using the following equation:

$$Error\ (\%) = \frac{The\ difference}{Average\ thickness} \text{ x } 100 \qquad \text{Eq (4)}$$

3. Discussion

The values of the difference between the coating thicknesses measured by the two methods as depicted in table 1, are too insignificant compared with the average coating thickness value. But the process of the two methods is totally different, the weight gain method is very simple and just needs to weigh the sample before and after coating. In contrast, the cross-sectional based method is a complicated technique since it needs more processing and equipment such as cutting machine and optical microscopy as well as time. Additionally, the most important thing is the resulting damage to the sample which prevents it from further usage for other processes.

Figure 1 shows that, the electroplating copper coating distribution is uniform; therefore, the weight gain method can be used to measure the thickness. The graph in figure 2 illustrates the comparison between the thickness values which were determined by weight gain as non-destructive and cross-sectional as destructive method. The thickness values are in the same range, but the difference between the values measured by the two methods increases with the increase in the time of coating, which indicates that, the difference increases with increasing thickness of the. In the same way, figure 3 explains the increasing of percentage error with increasing of time as well as the thickness/ increasing time as well as thickness, where the maximum error in percentage is 5.27% after 100mins and the minimum error in percentage is 4.11% at 20 mins interval.

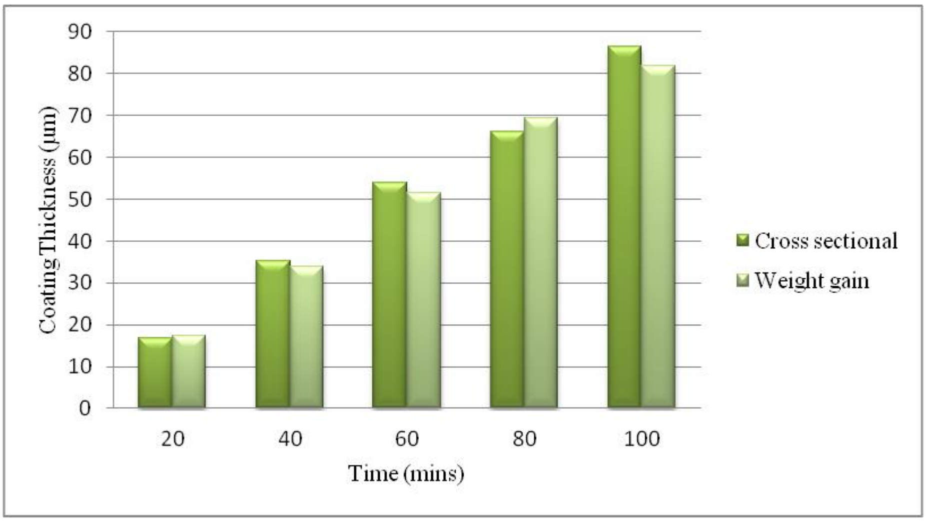

Figure 2: Comparison between the thickness values which determined by weight gain and Cross-sectional methods.

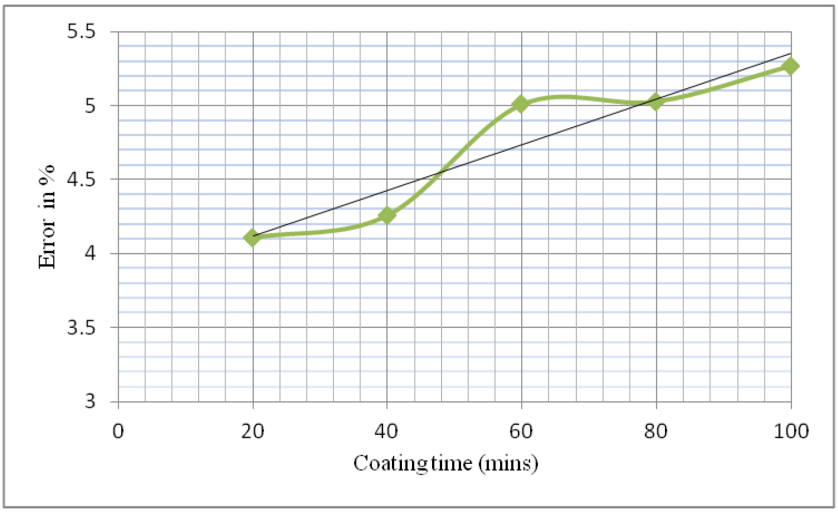

Figure 3: The error percentage between the two methods.

4. Conclusion

The results show that the thickness values measured by the weight gain method are as accurate as a cross-sectional method, with a small error of around 4.11% at coating time interval of 20 mins with a difference of 0.7 µm, this error increases with the increase in coating time and due to increasing coating mass. However, this error is still non-significant since its values is around 5.27% and the difference between the two methods values is 4.43 µm at 100 mins coating time. For this reason, the weight gain as non-destructive technique is more interesting than the cross-sectional method as destructive one since all samples can be tested without any damage. On the other hand, there are some limitations of weight gain method like, it can be used only with small parts and it is difficult to relate the mass of the coating to thickness if the substrate is rough or the coating distribution is not uniform, also information regarding the uniformity, porosity and the presence of cracks cannot be obtained. Likewise, the weight gain as non-destructive method is the simplest method that can be used to determine the coating thickness. In contrast, cross sectional based technique is more complicated due to the time and process it takes.

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