Non-Destructive Measurement of Metal Thickness in Displays Using Energy Dispersive X-Ray Spectroscopy (EDS)

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Abstract

Previously various optical, electrical, scanning, and destructive methods have been used to measure layer thickness. However, when measuring metal thickness, such methods either lack optical transmission, cause surface damage, or require sample destruction. Such drawbacks brings limitation in measurable sample type, loss of measured sample, and loss in overall production yield. Therefore, a non-destructive method for measuring metal thickness is essential for minimizing sample loss and maintaining production yield. In this study, an energy dispersive X-ray spectroscopy (EDS) was proposed as a non-destructive and sensitive method for measuring metal thickness. Based on the results of EDS, the metal thickness was calculated using intensity analysis method or ratiometric analysis method. The feasibility was first confirmed through Monte Carlo simulation for various metal types and thicknesses. The proposed method was further tested with copper and silver metal layer sputtered on glass substrate. Both the intensity and ratiometric analysis method positive linear relation as a function of metal thickness. However, the intensity analysis method showed slight difference in absolute intensity value between the measured modules, possibly due to the relative difference in sensitivity. The ratiometric analysis method, which calculates the ratio of the target to a secondary material, could compensates the sensitivity variation and showed equal and applicable results regardless of the detection modules. The results were further compared to conventional sheet resistance, and showed high sensitivity for all thickness. In conclusion, the proposed method is a sensitive and reliable measurement method of metal thickness and may a solution for non-destructive measurement in various fields of semiconductor, display, and battery.

Author Keywords

Metal thickness measurement; Non-destructive measurement; Metrology; Energy Dispersive X-ray Spectroscopy (EDS); Production Yield; Monte Carlo Simulation

1. Introduction

Previously various optical, electrical, scanning, destructive methods have been used to measure layer thickness, however they are not applicable when measuring the thickness of metal layer. Optical such as ellipsometery [1], reflectometry [2], WSI [3] have limited transmittance for metal. Electrical methods such as sheet resistance [4] can measure the lateral resistance, a representation of thickness, however the probes cause irreversible damage to the sample surface thus is not suitable for measurements in active pixel area. Destructive methods such as FIB-SEM can directly measure the thickness from the cross-section, however it requires sample destruction and loss in production yield. Therefore a non-destructive method for measuring the metal thickness is crucial for maintaining the production yield.

This paper suggests a non-destructive method for measuring metal thickness using energy dispersive X-ray spectroscopy (EDS). EDS is an analytical technique used for elemental analysis or chemical characterization of a sample [5]. It relies on the fundamental principle that each element has unique atomic structure allowing unique electromagnetic emission spectrum. The intensity of a specific element is dependent on the amount of the element present. Therefore, EDS could be utilized as a tool for measuring thickness based on the measured intensity. The feasibility was first tested through simulation and was confirmed with various thickness thin metal layer samples.

2. Materials and Method

Thickness Calculation

The thickness of the metal layer can be measured by either intensity analysis or ratiometric analysis method. One of the key parameters during EDS measurement is the interaction volume (or penetration depth), which depends on the accelerating voltage of the electron beam and the atomic number of the sample. The interaction volume is proportional to acceleration voltage, but is inversely proportional to the atomic number of the measured sample. When measuring thick metal layer sample, the volume percentage of the target metal would be large thus emitting strong EDS signal. Furthermore, it can be speculated that the intensity of the target EDS signal would show a linear and proportional relationship as a function of thickness. The ratiometric analysis is based on the intensity ratio of a target metal to a secondary material or a bulk target sample. Through ratiometric analysis possible differences between the sensitivity could be eliminated.

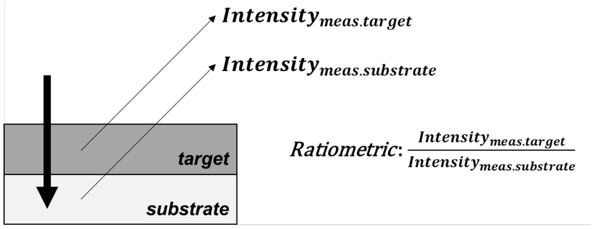

Figure 1. Schematic of intensity analysis and ratiometric analysis for thickness measurement using EDS.

Simulation

The feasibility of the suggested method was simulated through Monte Carlo simulation CASINO [6], and DTSA. The results of quantitative intensity simulation are shown in Figure 2. The generated intensity of single silver layer and underling glass (SiO_2) substrate showed opposing relationship as a function of thickness (Figure 2(a)). The signal intensity of silver increased while the silicon of the substrate decreased as the silver thickness

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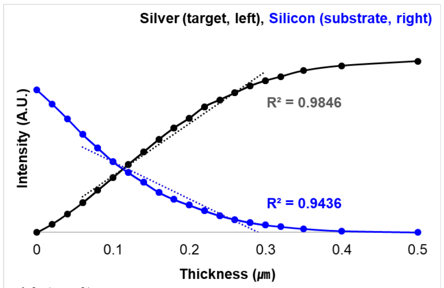
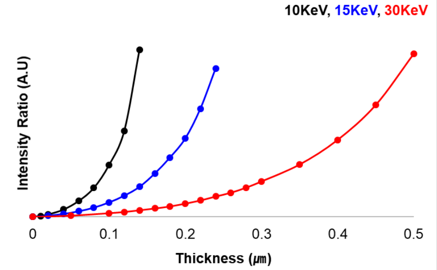
increased. The change in intensity were linear at the beginning, but reached a saturation region near 0.5 ㎛ thickness where the change in intensity were less significant. This suggests that there is a limitation in the maximum penetration depth at around 0.5 ㎛ thickness. It can be assumed that for sensitive and accurate measurement the thickness within the linear region should be used. Linear regression was calculated using the 15% – 85% intensity region, and showed good relation of R^2=0.96 (on average) suggesting that the selected region is suitable for thickness measurement. However, such intensity analysis methods would face a problem when the data require versatility (compatibility between different detectors). In reality, the two different detectors (same specification, same part number) cannot have identical sensitivity (detector sensitivity difference, alignment offset, source difference), thus the intensity analysis method may not be used alternatively between the detectors. Therefore, a compensation method eliminating the sensitivity differences between the modules is need. A ratiometric analysis method, which can compensate for sensitivity differences, is simulated for silver layer at various acceleration voltages (Figure 3(b)). Here, the ratiometric analysis was performed by calculating the intensity ratio of the target to a secondary material in the substrate. The results showed nearly linearly increasing region, followed by an exponentially increasing region regardless of the acceleration voltage. The range of the initial linear region were longer at higher acceleration voltage, suggesting that thicker layers could be measured with sensitivity.



a) Intensity



b) Ratiometic (Ag/Si)

Figure 2. Monte Carlo simulation of (a) intensity analysis method and (b) ratiometric analysis method. Linear regression in (a) was calculated using 15% – 85% of maximum intensity region.

Sample Preparation

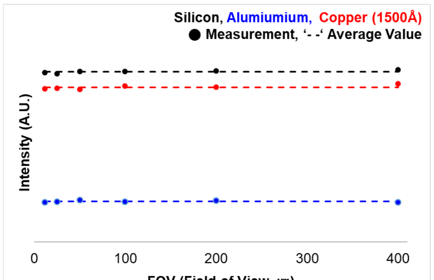
Thin layers of copper and silver layer were prepared on glass substrate using sputtering process. The metal thickness was controlled by adjusting the sputtering time to acquire target thickness of 250 Å, 500 Å, 1000 Å, 1250 Å, 1500 Å

EDS Measurement

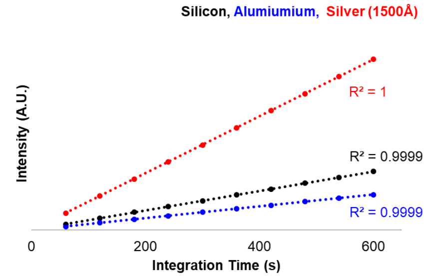
The prepared copper and silver samples were measured in a vacuum condition equipped two different Quantax 200 (Bruker) for EDS measurement. The measurements were performed in a vacuum condition equipped with cryopumps. The EDS was measured at 15keV and the generated X-ray signal intensity was measured and calculated using ZAF or φ (ρz) method

EDS Module Evaluation

Prior to the experiment, the performance of the EDS module was evaluated based on field of view (FOV) and integration time as shown in Figure 3(a) and Figure 3(b), respectively. The FOV evaluation and integration time evaluation was evaluated with 1500 Å copper and silver 1500 Å sputtered sample, respectively. The intensity of major composing materials; copper & silver (target), silicon & aluminum (substrate) are shown in the different colors. Figure 3(a) shows similar material intensity regardless of FOV showing that the source-detector alignment is good condition. The coefficient of variation (statistical measure of the dispersion of data around the mean) of the representative materials (copper, silicon, aluminum) were approximately 1.4% on average. Figure 3(b) shows linearly increasing intensity as a function of integration time. The integration time and measured intensity showed perfect linear relation (R^2=0.9999 on average), which implies that the modules are in stable condition even for long term measurement. In this paper, EDS was measured with FOV of 200 ㎛ and integration time of 200 s.



a) FOV Evaluation



b) Integration Time Evaluation

Figure 3. Evaluation of EDS module as a function of (a) field of view and (b) integration time. 'Dashed line' in (a) shows the average intensity value of each materials.

3. Results

Intensity Analysis

The proposed method was tested using copper and silver sample.

Figure 5(a) and Figure 5(b) show the change in EDS intensity of copper and silver sample as a function of target thickness. EDS signals of the target material and underlying substrate materials are shown in Figure 4(a1, b1) and Figure 4(a2, a3, b2, b3), respectively. Measurement from individual modules were separately depicted in red and blue. In general, as the metal thickness increased the intensity of the target increased, while the intensity of the substrate decreased. Both the increase in the intensity of target metal signal and decrease in the intensity of substrate material showed linear relation of R^2=0.98 on average. However, it can be noted that the measurements of the Mod.1 and Mod.2 show similar increasing trend but with slightly different intensity. Mod.1 (Red) showed approximately 20% higher EDS intensity compared to Mod.2 (Blue), and were similar regardless of the material or thickness. Such difference in signal intensity could be due to the relative difference in sensitivity possibly caused by the difference in detector sensitivity, H/W alignment, and H/W characteristics. Because of such differences the results acquired from one module could not be directly used with another module, and thus needs further compensation.

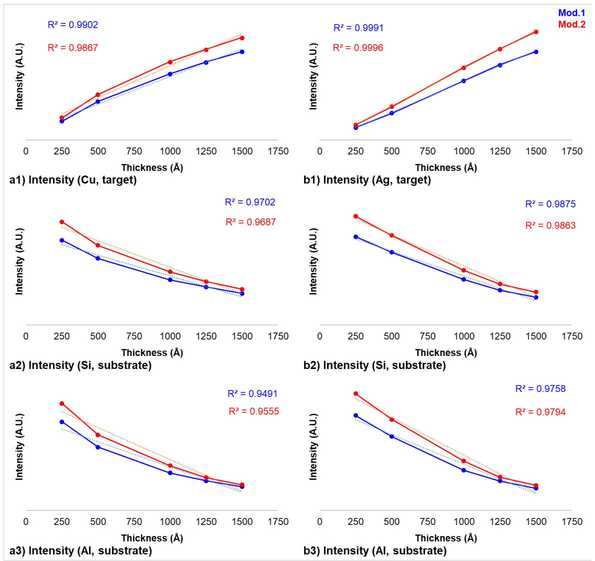


Figure 4. EDS measurements of (a) copper sample and (b) silver sample.

Ratiometric Analysis

The measured data were further processed to compensate the relative differences between the modules. A ratiometric analysis were performed by calculating the ratio of the target to a secondary material of the substrate (Silicon and Aluminum). Figure 5(a1, a2) shows ratiometric results of copper sample compared with silicon and aluminum, respectively. Figure 5(b1, b2) shows ratiometric results of silver sample compared with silicon and aluminum, respectively. Through ratiometric analysis a perfect correlation between EDS modules. All the results showed a good linear regression of R^2=0.95 on average. It can noted that despite the difference in signal intensity, of approximately 3 times, between silicon and aluminum, (Fig 4(a2, a3) and Fig 4(b2, b3)), the result trends were similar to the other. The difference between the results of each module are approximately 1%, proving that the ratiometric analysis can compensate for the difference of the modules and the results can be interchangeably used.

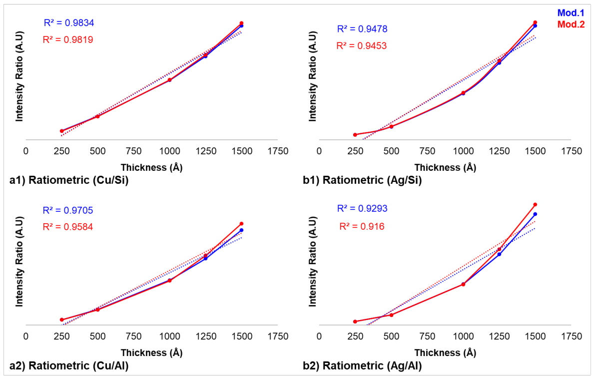


Figure 5. Ratiometric analysis of (a) copper and (b) silver sample. Ratiometric analysis performed using (a1, b1) silicon, (a2, b2) aluminum.

Comparison with Sheet Resistance

The results of the proposed method were compared to the sheet resistance, commonly used method for measuring metal thickness. Figure 6(a) and Figure 6(b) shows the average sheet resistance (black) and EDS Measurement (blue) of copper and silver sample, respectively. For both copper and silver, the sheet resistance showed exponential decay as the thickness increased, while the EDS signal showed linear increased. The results of sheet resistance were within expectation where thicker layers would have higher electron conductivity, and thus lower sheet resistance. In the relatively thicker region (of 1000 Å and above) the change in sheet resistance was less significant compared to thinner regions (250 Å – 500 Å). Such exponential decay in sheet resistance may bring difficulty in sensitive discrimination of sample's thickness. However, the EDS measurement method showed linear relation regardless of the target thickness, which implies that the EDS measurement method is more intuitive for thickness measurement.

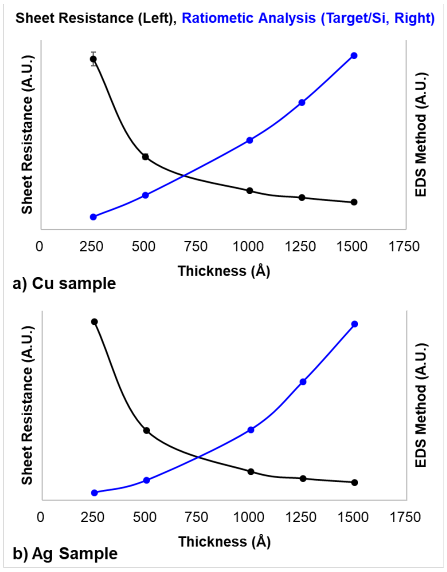


Figure 6. Comparison between (black) average sheet resistance and (blue) Ratiometric method for (a) copper and (b) silver deposition sample. Ratiometric analysis were performed with Si.

Figure 7(a) and Figure 7(b) shows the comparison between sheet resistance (black) and EDS Measurement (blue) of copper sample, respectively. There was slight variation in sheet resistance, suggesting a variation in the metal thickness within the sample. The EDS method showed inverse relation to the sheet resistance, where high resistance (thin thickness) showed low EDS value. The proposed method showed good correlation of R^2=0.93, suggesting that the EDS method shows similar sensitivity compared to the conventional sheet resistance.

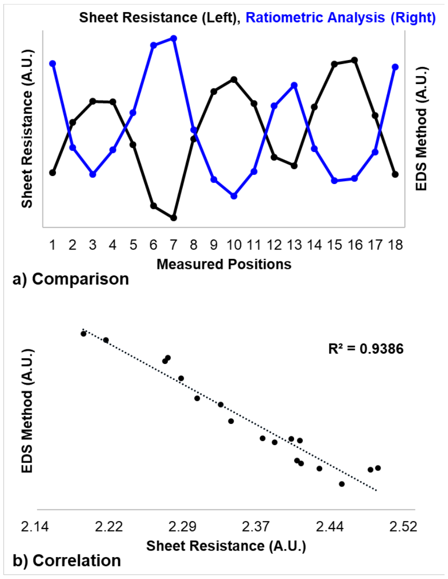


Figure 7. (a) Comparison between individual (black) sheet resistance and (blue) EDS measurements of 250 Å silver sample. (b) Scatter plot of the sheet resistance and EDS measurements.

4. Discussion and Conclusion

In this study, EDS was used as a non-destructive method to measure the metal thickness. Two different analysis methods (intensity analysis and ratiometric analysis method) were proposed and they were confirmed through simulation and experiment.

The feasibility was first confirmed through simulation showing increase in target intensity and decrease in substrate intensity as a function of target thickness. Based on the simulation results, copper and silver samples were prepared and measured using EDS. The intensities of copper and silver showed positive linear relation to their thickness, while the signal of the substrate showed negative linear relation to target thickness. There was slight difference in the acquired intensity between the two modules, which could be due to relative difference in sensitivity or alignment between the modules. With the ratiometric analysis method, the measurements of two different EDS showed identical results suggesting that the sensitivity factor was removed. The EDS measurements were further compared to conventional sheet resistance results. Conventional sheet resistance showed exponential decrease, whereas EDS measurement showed linear relation to the thickness suggesting that the EDS method is sensitive for both thin and thick samples. The results showed that the proposed method is sensitive to the metal thickness and may a solution for non-destructive measurement.

5. References

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