DETERMINATION OF THE THICKNESS OF GOLD LAYER

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A method for the determination of the thickness of gold layer on top of silver substrate has been developed. Various samples having silver as the substrate were coated with gold for different time durations by ion sputtering. The gold to silver X-ray counts ratios were determined using an energy dispersive spectrometer equipped to the scanning electron microscope. A graph plotted between the gold to silver X-ray counts ratios and the thickness of the gold layers enables to determine the gold layer thickness of an unknown sample by merely measuring its gold to silver X-ray counts ratio and then extrapolating this ratio to its corresponding thickness.

Key words: Gold layers, Silver substrate, Ion sputter.

Introduction

Most medals, trophies and decorated articles consist of a silver (Ag) substrate with a gold (Au) plated layer on top. Typically the thickness of this Au layer is measured directly. However, this posed a problem due to inadequate Scanning Electron Microscope (SEM) resolution and the inability to focus the cross section of the sample sufficiently well on the optical microscope. In order to overcome these problems, we report here a new method for the determination of Au thickness on Ag substrate. The method can measure coating thickness as of 1µm. It has the added advantage that it is a non-destructive method, provided that the sample size does not become a limitation for the SEM chamber.

Experimental

Ag sheet samples having a thickness of 2mm were punched to obtain samples for Au coating. These were weighed before being Au coated under vacuum. Au coating was performed using the ion sputtering technique for periods ranging from 10 to 60 minutes in steps of 10 minutes. The current value on the Au sputter was set at 10mA by regulating the Argon gas pressure, and the voltage was maintained at 1.2 kV. The distance and angle between the samples (anode) and the Au cathode were kept constant.

After the coating process was complete the Au samples were reweighed so as to obtain the mass difference for the Au layer deposited on each sample. Thickness values for the Au coated samples were calculated for the corresponding time durations. The samples were then analyzed using an Energy Dispersive Spectrometer (EDS) at 20 kV.

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Results and Discussion

Table 1 shows the change in mass of the samples for the corresponding durations of Au coating.

Table 1

Au coating time (min)	10	20	30	40	50	60
Wt of Au layer (µg)	55	110	165	220	275	330
Thickness of Au layer (µm)	0.05	0.10	0.15	0.20	0.26	0.32

The thickness values were derived as follows: -

$$\rho = \frac{m}{v} \quad \text{............} \quad (1)$$

where 'ρ' is density, 'm' is mass and 'v' is volume

$$\text{Also} \quad v = \pi t r^2 \quad \text{or} \quad \frac{\pi t D^2}{4} \quad \text{............} \quad (2)$$

where 't' is thickness and 'D' is diameter

$$\text{From (1) and (2) above} \quad t = \frac{4m}{\pi D^2 \rho} \quad \text{............} \quad (3)$$

Equation (3) was used to calculate the thickness values in Table 1.

EDS spectra of Au coated samples were also taken. The X-ray counts for Au/Ag X-ray counts ratio were determined after subtracting the background contribution. It may be noted here, that the windows for the Au and Ag peaks were set at the same energy levels for all the samples. The Au/Ag X-ray counts ratio thus obtained was plotted against time (Fig 1).

With the help of Fig 1 and Table 1 a graph was plotted with Au/Ag X-ray counts ratio as a function of thickness (Fig 2).

The interaction of electron beam with the specimen may be understood with the help of the schematic diagram in Fig. 3. As may be seen, the X-rays emitted from the specimen do not arise from the surface of the metal alone. Instead the signal is also obtained from a bulb like region beneath the surface. These X-rays are further absorbed by the material before being detected by the EDS detector which is placed at a take off angle of 35° to the sample. The higher the kV, the greater the penetration depth of the electron beam and the higher the atomic number the smaller the bulb size. (Goodhew and Humphreys 1988 ; William David 1984).

According to the plot in Fig.2, the thickness of Au onto the Ag substrate varies with the Au/Ag X-ray counts ratio in three distinct regions. These are denoted by A, B, and C. In region A, the shape of the bulb is as shown in Fig.4a. A very thin region
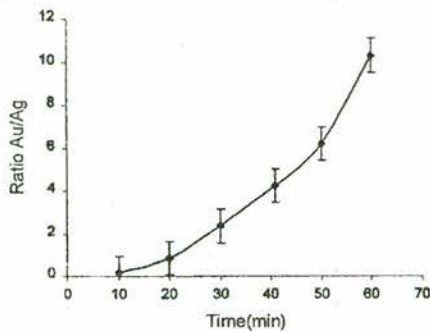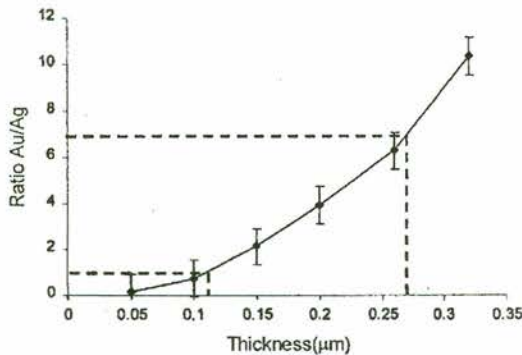

Fig 1. Aplot of Au/Ag ratio vs. time (min).

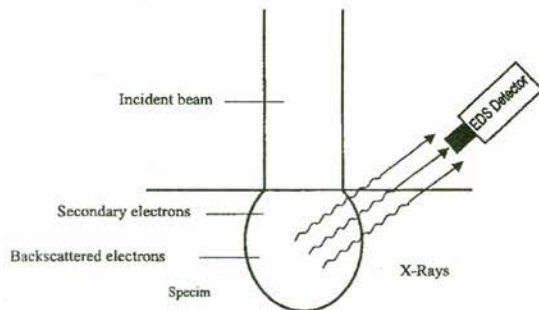Fig 2. Plot showing relationship between Au/Ag x-ray counts ratio and Au thickness (μm).

Fig 3. Schematic sketch showing interaction of electron beam with specimen.

below the surface is Au while the Ag substrate is relatively very thick. The X-rays emitted from the sample are mostly from the Ag substrate and the effect of Au coating is negligible. The spectrum obtained in this case is also shown in the same figure. For the same kV, this effect is different for region B (see Fig 4b). Here, the contribution of both Au and Ag is equally significant. The bulb formed beneath the surface of the specimen has the same proportion of Au and Ag. The specimen also shows the presence of Au and Ag peaks in almost equal proportions. This region corresponds to a Au/Ag X-ray counts ratio ranging between 1 and 7. In region C, the bulb assumes the shapes as in Fig 4c. The Au coating being thicker as compared to Fig. 4a and 4b, the signal obtained is mainly from Au with only a small amount of X-rays obtained from the Ag substrate. Moreover, Au being the heavier of the two metals, most of the Ag X-rays are absorbed by it. As a result, a very small number of Ag X-rays are detected.
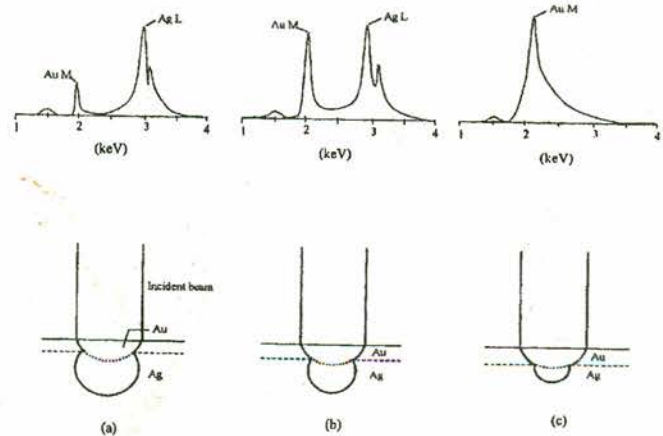

Fig 4. Diagram showing the effect of Au coating thickness in regions A, B, and C of Fig. 2. The EDS spectra corresponding to the three regions are also shown.

Conclusion

Au coating thickness on Ag substrate may be determined for Au/Ag X-ray counts ratio value ranging between 1 and 7.

The method is non destructive.

Acknowledgement

References

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