Study and comparison of deposition rates, grain size of Ag and Cu thin films with respect to sputtering parameters, and annealing temperature.

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Abstract: Deposition rates of Ag and Cu thin films with respect to sputtering parameters have been compared. Discharge current range of 200-1200 mA, gas pressure (Ar) with range of 0.02-0.2 mbar has been used. Dependency of rate, on current gas pressure and horizontal and also vertical distance has been studied. Deposition rate for copper was 0.5-2.8 nm/s while for silver films was 1.8 to 7.9 nm/s, and thickness of films was 135-1400 nm and 20-150 nm respectively. By help of curve fitting these results have been leaded to an empirical relationship. X-Ray Diffraction (X.R.D) of these thin films have been obtained for various thickness and deposition condition as well as different annealing temperature in argon flow oven (up to 500 °C) and peak (111) of Cu and Ag which were more pronounced was chosen for investigation and its variation with different coating condition have been carried out for grain size studies.

1. Introduction
Since both silver and copper are widely used as thin films in micro-electronic circuits, due to their low electrical resistivities, as well as being stable elements, (specially silver) and also both of these (fcc, Nobel/transition) metals are used in optic and lasers for making metallic mirrors, since their optical reflectivity in visible and infra-red is very high [1,2].

Film thickness, deposition rate, and method of coating play important role on microstructure as well as macroscopic properties of films [3,4]. Also deposition rate is function of various factors. In this work for above reason, dependency of deposition rate on sputtering parameters (discharge current, gas pressure, distance between target and substrate) have been investigated. Effects of substrate annealing temperature on different properties also were studied. X-Ray diffraction (X.R.D) of produced thin films was carried outs, and grain size and variation of it with respect to deposition conditions was investigated, and different obtained results for silver and copper were compared [5,6]. These two elements have similar physical and chemical properties, and this is the reason they were chosen for comparison.

2. Experimental details
For exact knowledge about deposition rate and how it affects the various properties of Ag and Cu thin films, a vacuum plant with base pressure of 10⁻⁷ mbar was employed. Silver and copper targets were made with high purity and used for magnetron sputtering system. Also high purity Argon (99.999%) was employed for plasma and discharge formation, and pressure range of 0.02-0.2 mbar was used. Distance between target (cathode) and glass substrates was changed from 5 to 15 cm, and discharge
current was increased up to 1000 mA, and voltage due to variation of gas pressure and current to sustain the discharge was varied as well.

To measure thickness and deposition rate of Ag and Cu, a digital vibrating quartz crystal as well as an exact balance (10\(^{-4}\) gr) were employed to double check the results. Glass substrates were used and just before using them, they were washed and cleaned ultrasonically in heated acetone. To monitor temperature of substrates during deposition and after that, an exact digital thermocouple was employed and was placed near the glasses on substrate holder. A moveable shutter was placed between target and glass substrate for two reasons: first to make sure of purity which was observed by a spectrometer with high resolution power, and plasma was checked for line spectra’s of carrier gas atoms, ions and material of target, secondly to control the period of deposition exactly. To study the microstructure of produced thin films in this work, X-Ray Diffraction (X.R.D) of them were carried out, and effect of various sputtering parameters was investigated. For annealing the samples, an Argon flow oven was employed, which could be heated up to 1000 °C.

3. Results
To study the effect of sputtering parameters on deposition rates of silver and copper, were Ar pressure was kept constant (0.02 mbar), both showed a linear behavior with electrical power (\(P_e\)) (discharge current time required voltage). Figure (1) shows variation of deposition rate on center of coating (\(\hat{R}\)) against electrical power, as it can be observe, gradient for Ag is higher than Cu. Since at constant gas pressure and fixed distance between target and substrate, \(\hat{R}\) is related directly to electrical power, we have equation (1) [2-4]:

\[
\hat{R} = K \frac{Vi}{Pd} = K \frac{P_e}{Pd}
\]

where \(P\) is gas pressure and \(d\) is electrodes distance, \(K\) being constant of proportionality, which is function of type of gas that is used (sputtering ion) and material of target. \(V\) and \(i\) are voltage and current respectively. When distance current was kept constant at 0.6 A and distance between targets (Ag & Cu) and substrate was also 12 cm, variation of \(\hat{R}\) against Ar pressure is given in Figure (2). In both Figures (1, 2), deposition rate of Ag is roughly twice the copper rate for similar experimental conditions.

![Figure 1: Variation of deposition rate on center (\(\hat{R}\)) against electrical power.](image1)

![Figure 2: Variation of \(\hat{R}\) against Ar pressure](image2)

To study the microstructure of these produced thin films, X.R.D. of copper thin films for various annealing temperatures in argon flow oven were obtained. Figure (3) shows X.R.D. pattern of Cu for different annealing temperatures. Since peak (111) for both Ag and Cu was much more intense, measurements were carried out on this peak to reduce the error.
In pattern of X.R.D. of Ag thin film, again since peak (111) of Ag (the same as Cu) is more pronounced. All measurements were concentrated on this peak and its variation with deposition parameters was investigated. With using Scherrer equation [5]:

\[ D = \frac{A\lambda}{B\cos\theta} \]  

where \( D \) is grain size, \( B \) is full half width of peak, \( \lambda \) is wavelength of X-Ray, \( \theta \) being bragg angle, while \( A \) is proportionality constant, which usually have value near to unity, and depends on crystallites and their distributions [6].

![Figure 3: X.R.D. pattern of Cu thin films for various annealing temperatures in argon flow oven.](image)

In Figure (4) variation of Ag (peak (111)) W.R.T different discharge current is given. Figures (5, 6) show how gas pressure (Ar) and vertical distance affects this peak of Ag. In Figures (5, 6) one can get this result, when deposition rate is higher, height of (111) peak of Ag increases. By measuring grain size of Ag and plotting variation of grain size against deposition rate, we can see as current rises, grain size reduces.

![Figure 4: Variation Ag (peak (111)) for different discharge current.](image)  

![Figure 5: Variation Ag (peak (111)) for different pressures.](image)
4. Summary and conclusion

- Deposition rate of Ag in similar condition is higher than Cu.
- Dependency of Cu deposition rate on Ar pressure is more than Ag.
- In lower distances, deposition rate of Ag is much more than Cu, but for higher distances they tend to a similar amount.
- Annealing temperature affect the X.R.D. of Cu, at higher temperature more peaks were obtained, after temperature of 400°C, variation of (111) peak of Cu was very little.
- By increasing current, or decreasing gas pressure and vertical distance (h), which in all cases deposition rate increases, peak (111) of Ag becomes more pronounce.
- By measuring grain size, we see when deposition rate increases, grain size show reduction.

Acknowledgment
This work was been partly supported by nano-technology initiative of Iran, the authors are grateful for their support.

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