The impact of deposition time on the morphological and structural characteristics of silver nanoparticles using the DC sputtering process.

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Abstract. Silver nanoparticles were prepared on glass substrates at room temperature using four different deposition times (10, 15, 20, and 25 minutes). The dc magnetron sputtering technique is used to prepare the surface, with thicknesses of (30, 40, 50, and 60) nm, respectively. The effect of deposition times on the microstructure and morphology of the surface of deposited thin films was examined. Moreover, X-ray diffractometer (XRD) and atomic force microscopy were used to examine the structural properties and morphological characterization of sputtered films. Parameters such as crystallite size are also measured. The research indicates that the XRD pattern has a polycrystalline structure with a preference for orientation along the (111) axis. The AFM images confirmed that the thin films shaped uniformly distributed spherical particles (in terms of size). Finally, increasing the film thickness causes the average surface roughness of the films to increase between (2.6-11.6) nm.

Keywords: DC Sputtering; Crystal growth; thin film, surface roughness, Surface morphology.

1. Introduction

The creation of noble metal nanostructure strategies with controlling the form of surface structures fully, remains a major challenge [1]. New advances in functional materials have been introduced as a result of the growth of nanotechnology and nanoscience. Owing to the particular value of metallic thin films in a number of application areas, Materials' physical properties change as well. as it shrinks in scale [2]. for years, silver has sparked the attention of metallic thin film manufacturers due to its superior performance over other metals [3] Weight, malleability, ductility, and high electrical and thermal conductivity are some of its mechanical properties. It's also known for its ability to withstand extreme temperature changes. Nanoparticle films with a high level of Plasmon polarization [4,8] resonance in sensor applications, they are used as optical filters. Thin gold films' surface microstructure is considered to be highly dependent on thin-film deposition parameters [9], this allows them to vary their quality. Metal oxide, nitride, and carbide films, as well as thin films, nanostructured coatings, and nanoparticles, have all been generated using direct current magnetron sputtering [10–16]. DC magnetron sputtering may also be a viable method for producing metal nanoparticles. The technique also improves the efficiency of synthesis processes and improves control over the scale, shape, and aggregation of nanoparticles [17].

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The magnetron sputtering plasma technique's efficiency makes it a viable option for metallic nanoparticle synthesis, due to the lack of issues associated with other approaches [18]. The aim of this research is to see how the deposition time of Ag nanostructures by magnetron sputtering affects their properties. The influence of deposition time on the microstructure and morphology of thin film surfaces is studied. The structure that was created was characterized by an X-ray diffract meter (XRD), atomic force microscopy (AFM), to gain a comprehensive understanding of the nanostructured substrates that have been created.

2. Experimental Procedures
2.1. Material Preparation
On glass substrates, silver thin films were deposited using a DC magnetron sputtering system. This system is made up of a 99.9% Ag-metal planar electrode with a diameter of 5 cm. The DC glow discharge device consists of two electrodes in a cylindrical chamber. The cathode is facing the anode, which creates an electric field for the gas discharge. When the distance between two electrodes is 4 cm and the flow rate of argon gas is 40 sccm, discharge occurs. A rotary pump was used to evacuate the container. (Edward 12 m$^3$/h). A flowmeter was used to restrict the amount of gas inside the chamber, and a Pirani gauge with an Edward controller was used to measure the pressure inside the chamber. A DC-power supply was used to provide variable applied voltage. A limiting resistor was used in the circuit to keep the discharge below the abnormal glow discharge field. Multi-meter optical measurement of discharge potential and discharge current.

The films were formed at ambient temperature in a deposition chamber and evacuated to a base pressure of $2 \times 10^{-1}$ mbar, and then the steady Ar working pressure was maintained according to a throttle valve that settled of $3 \times 10^{-1}$ mbar. The film was deposited at 630 V in a constant voltage setting. Deposition took place at various times, the films were deposited: (10, 15, 20, and 25 min). The thin film thickness was determined with a 45-degree incident angle using a He-Ne laser (0.632 m) wavelength optical interferometer device. The interference of the laser beam transmitted from the thin film surface is used in this technique; using the formula below, the film thickness ($t$) was calculated [19].

$$t = \frac{\lambda}{2} \cdot \frac{\Delta x}{x} \quad (1)$$

Where $x$ is the fringe width, the difference between the two fringes is $\Delta x$, and $\lambda$ is the wavelength of laser light.

2.2. Characterization
X-ray diffraction was used to examine the structural characteristics of Ag nanoparticles. The surface topography of the films was analyzed using atomic force microscopy. AFM (atomic force microscopy) is an excellent tool for studying the morphology and texture of various surfaces. The ability to examine complex biological processes has been made possible by understanding surface topography with Nano metric resolution. [20].

Average roughness, mean square roughness of root, are used to perform a detailed study of Ag film surface properties. These parameters provide information about the surface's characteristics and efficiency.

3. Results and Discussions
3.1. Structural characterization
The XRD patterns of Ag nanoparticle thin films measured in this study are shown in figure 1. It can be shown that at $2\theta = (38.28^\circ)$, the dominant peak matches (111) silver plane, indicating a clear preference for this direction. The diffraction peaks of samples observed correspond to Ag face center cubic structure (FCC) according to (card No. 7440-22-4) [21]. The Scherrer formula was used to calculate the crystallite size for various deposition times, which is given by [22]:

$$D = \frac{K \lambda}{\beta \cos \theta} \quad (2)$$

Where $D$ is the size of the crystallites, $K$ is a constant, $\lambda$ is the wavelength of the X-ray, $\beta$ is the full width at half maximum of a peak, and $\theta$ is the Bragg angle.
\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \] (2)

Where the wavelength is \( \lambda \) of the x-ray, \( \beta \) is the distance at half maximum intensity (FWHM), and \( \theta \) is the angle of Bragg. Table 1 displays the structural property parameters.

**Table 1.** Experimental value (card No. 7440-57-5) of dhkl for the (Ag) peaks shown for different sputtering times in XRD

| Deposition time(min) | 2\(\theta\) (deg) | FWHM (deg) | \(d_{hkl}\) | Exp. (A) | C.S (nm) | hkl | Thickness (nm) |
|---------------------|-----------------|------------|-------------|----------|----------|-----|----------------|
| 10                  | 38.19           | 0.7475     | 2.35415     | 10.35    | (111)    | 30  |                |
|                     | 44.09           | 0.42500    | 2.05214     | 18.20    | (200)    |     |                |
| 15                  | 38.13           | 0.6345     | 2.35794     | 12.19    | (111)    | 40  |                |
|                     | 44.05           | 0.43221    | 2.04998     | 17.90    | (200)    |     |                |
| 20                  | 38.18           | 0.50630    | 2.35478     | 15.28    | (111)    | 50  |                |
|                     | 44.30           | 0.44170    | 2.04298     | 17.52    | (200)    |     |                |
| 25                  | 38.22           | 0.55450    | 2.35251     | 13.95    | (111)    | 60  |                |
|                     | 44.34           | 0.63750    | 2.04088     | 12.13    | (200)    |     |                |

3.2. Surface morphology
A microscopic analysis of atomic force is ideal for quantifying nanoparticle dimensional surface roughness and visualizing the surface Nano-texture of prepared films. The histogram and three-dimensional (3D) AFM images of different deposition times of Ag films: (10, 15, 20, and 25 min.) are
shown in figure 2. The roughness on the surface of Ag films increased as the deposition times increased from 2.684 to 11.62 nm. After 10 minutes, the Ag coating layer can be seen in the homogeneous structure. At 25 minutes, the various particles agglomerate in Small Island to form a new sheet. With raising thickness, the grains grow larger and may eventually form clusters that combine these clusters to form large grains, implying that the thickness increases with increasing sputtering time, resulting in increased average grain size, average roughness, and root mean square roughness. The roughness of the surface was determined by calculating the roughness parameters Rq, Ra, and Maximal elevation. The parameters of the surface profile include average roughness (Ra), root mean square roughness (Rq). The average roughness (Ra) is the average height calculated over the entire measured length/area. Ra is most often used to describe the roughness of machined surfaces. It aids in the analysis of general variations in the overall height of the profile's characteristics, as well as the monitoring of an ongoing production process. The square root of the surface height distribution is defined by root mean square roughness (Rq). It is well established that it is more vulnerable to large deviations from the mean line/plane than average roughness.

![3D AFM image and histogram of coating layer Ag nanoparticles deposited at various sputtering times.](image)

**Figure 2.** 3D AFM image and histogram of coating layer Ag nanoparticles deposited at various sputtering times.
Table 2. Roughness parameters of Ag thin films deposited at different time.

| Deposition time (min) | Ra (nm) | Rq (nm) | Average grain size (nm) |
|-----------------------|---------|---------|-------------------------|
| 10                    | 2.684   | 3.849   | 19.31                   |
| 15                    | 7.728   | 9.582   | 28.96                   |
| 20                    | 4.659   | 6.229   | 37.21                   |
| 25                    | 11.62   | 14.36   | 52.79                   |

Figure 3 shows how the density of data points can be distributed on the surface using a depth histogram. Graduation of the vertical axis in-depth: There is a graduating of the horizontal axis in % to about the population as a whole. The Abbott-Firestone curve corresponds to the bearing ratio curve, i.e., for a given depth, the proportion of the substance crossed in relation to the area covered. This function is the amplitude distribution form's cumulative function. The bearings percent ratio % is represented on the horizontal axis. The depths are described by the vertical axis. (in the measurement unit).

4. Conclusion
The effect of deposition time on the structural and morphological properties of Ag thin films grown on glass substrates by DC magnetron sputtering at room temperature is investigated. Various characterization methods were used to analyze the impact of thickness variation in these films in great
detail. The XRD studies show how pure Ag samples with a cubic structure centered on the face formed. The surface of Ag Deposited Thin Films has a wavy surface texture, as shown by AFM topography the grain sizes of Ag thin films increase with increasing film thickness at different deposition times, according to XRD and AFM analysis.

With increasing deposition time, the film surface becomes rougher, and specific grains are discovered. This type of study provides a more in-depth understanding of the effect of deposition conditions on the morphological characteristics of films, and it can be used to adjust deposition parameters to the specifications of surface morphology for the application of an optoelectronic device.

Reference
[1] Ondrej S, Soňa F, Teodora I, Ivan N and Vladimír T 2016 Gold nanostructures sputtered on zinc oxide thin film and corning glass substrates Electrochim. Acta 59 88
[2] Pradhan S 2016 Observation of Optical Properties of Gold Thin Films Using Spectroscopic Ellipsometry J. Phys. D: Appl. Phys. 49 065011
[3] Gan Q, Ding J, Bartoli F 2009 “Rainbow” trapping and releasing at telecommunication wavelengths Opt. Express 17 12909
[4] Lazziri R and Jupille J 2011 Quantitative analysis of nanoparticle growth through plasmonics Nanotechnology 22 445703
[5] Maaroof A and Smith G 2005 Nanoporous plasmonic coatings. In Nanocoatings Thin solid films 206 485198.
[6] Sun X 2007 Influence of random roughness on the Casimir force at small separations Thin Solid Films 508 5156962
[7] Wei J and Gan F 2003 Time-resolved thermal lens effect of Sb thin films induced by structural transformation near melting temperature Opt. Commun. 219 261-269
[8] Lai Y, Zhang H, Sugano Y, Xie H and Kallio P 2019 Correlation of Surface Morphology and Interfacial Adhesive Behavior between Cellulose Surfaces: Quantitative Measurements in Peak-Force Mode with the Colloidal Probe Technique Langmuir 35 7312
[9] Liu Z and Brown N 1997 Studies using AFM and STM of the correlated effects of the deposition parameters on the topography of gold on mica Thin Solid Films 300 84
[10] Safi I 2000 Recent aspects concerning DC reactive magnetron sputtering of thin films: a review Surf. Coat. Technol. 137 203
[11] Mohammed K K, Baha A M, ALhilli b, Ausama I K, Anwar A 2016 Photonics and Nanostructures Fundamentals and Applications 1669 4410
[12] Alaa N 2011 Java Applet Technology for Design Interference Optical Coating Baghdad Science Journal 8 2
[13] Sheebal N, Namitha A, Ramsiya K, Ashitha T, Suhail R 2021 Effect of Deposition Time on Structural and Optoelectronic Properties of Flower-Like Nanostructured PbS Thin Films J. Sci. Res. 13 1
[14] Bock F, Christensen T, Rivers S, Doucette L and Lad R 2004 Growth and structure of silver and silver oxide thin films on sapphire Thin Solid Films 468 1
[15] Dmitry I, Aleksey V, Yury V, Dmitry Y and Valentyt S Optical constants and structural properties of thin gold films 2017 Optics express 25 21
[16] Hou S, Ouyang M and Chen H 1998 Fractal structure in the silver oxide thin film Thin Solid Films 331 322
[17] Korner E, Aguirre M, Fortunato G, Ritter A, Ruhe J and Hegemann D 2010 Plasma Processes and Polymers 7 619
[18] Khalaf, M. K., Al-alWany, R. M. S., & Salman, I. K. (2019). Effect of working pressure on the structural and morphological properties of gold nanoparticles prepared by a dc magnetron sputtering technique. Journal of Critical Reviews, 7(1)
[19] Khalaf, M. K., Mazhir, S. N., Mahdi, M. S., Taha, S. K., & Bououdina, M. (2018). Influence of RF sputtering power on surface properties and biocompatibility of 316L stainless steel alloy by deposition of TiO2 thin films. *Materials Research Express, 6*(3), 035401.

[20] Ruffino, F., & Grimaldi, M. G. (2018). Morphological characteristics of Au films deposited on Ti: a combined SEM-AFM study. *Coatings, 8*(4), 121.

[21] Krishnamurthy, S., Esterle, A., Sharma, N. C., & Sahi, S. V. (2014). Yucca-derived synthesis of gold nanomaterial and their catalytic potential. *Nanoscale research letters, 9*(1), 1-9.

[22] Mahdi M, Ibrahim K, Hmood A, Ahmed N and Mustafa F 2017 Control of phase, structural and optical properties of tin sulfide nanostructured thin films grown via chemical bath deposition *J. Electron. Mater. 46* 4227