Silver and Cadmium Mattel segment Doping by DC Sputtering

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Abstract:
This research deals with the process of de forming cadmium metal with silver metal directly by planting pieces of silver cylindrical shape on the surface of the target material of cadmium and by using the DC reactive magnetron sputtering technique deposit on a glass substrate. The flow rate of argon and oxygen gases fixed to (90 and 10) sccm and sputter time was 120 min per model (0, 1, 2 and 3) Ag chips, the current of sputtering and the voltage of sputtering were kept within the process of deposition at 18 mA and 1300 V, The temperature of substrate had been set at 150°C. The structural properties showed that all films prepared have a polycrystalline diffraction pattern with a predominant orientation of the plane (111) and an increase in the size of the crystal from 17.17 nm to 19.30 nm with the number increases of chips above the surface of target material, and the growing of spherical nanoparticles with a diameter ranges from (25-40) nm

Keywords: Cadmium Oxide, Sliver, Chips technique, Transition Electron Microscope, DC reactive magnetron sputtering.

Introduction

Silver was known to have antibacterial activity for a long period of time, and was used till chemical antibiotics came on the scene. Metallic silver and the most inorganic silver compounds ionize in humidity, body fluids and mucus to launch biologically active Ag+, which the silver ions used as an antimicrobial agent for the toxicological and pharmacological properties[1]. Silver nanoparticles (AgNPs) are of particular interest among different metals due to its excellent antimicrobial and localized surface characteristics Plasmon resonance. That is makes them remarkable characteristics, like antimicrobial broad-spectrum [2], biological /chemical sensors, materials of biomedicine, biomarker, surface-enhanced Raman spectroscopy (SERS) and so forth[3]. Silver nanoparticles normally range in size between 1 and 100 nm[4]. They possess remarkable electrical, optical and thermal characteristics and are integrated into electronics industrial applications, photonics, and catalysis[5]. The synthesis of AgNPs thus is an important topic in the electronic field. Silver nanoparticles (AgNPs) are widely used with strong antimicrobial activity as one of the main ingredient in industrial, daily and health-related products[6]. Due
to its applications in recent years, researchers have focused on cadmium oxide (CdO). In particular, for opto-electronic applications like cells of solar [7], transistors and photographic diodes, gas sensors, and transparent electrodes, so forth [8]. Cadmium oxide (CdO) is a rock-salt crystal structure (F.C.C) n-type semiconductor and has a direct band gap (2.2-2.5) eV[9].

Various technologies for preparing CdO thin films like activated reactive evaporation [13], solution growth [12] pulsed laser sputtering [14] spray pyrolysis [10], dc sputtering [15], sputtering[11] magnetron sputtering [16]and sol-gel method [17]. One of the best techniques among these is DC magnetron reactive sputtering. Because of its high deposition rates on a wide area it offers good control over the composition of the film deposited. The high rates of deposition minimize poisoning target that has a massive problem in reactive sputtering. Moreover, as its substrate temperature rise throughout deposition is low, the ratio of stoichiometry of the films can be easily controlled[18]. This method-Magnetron sputtering, can be explained as a gaseous plasma deposition technology that is created and confined to a space that contains the material that will be deposits – the 'target'. The target surface is corroded by ions with high-energy inside the plasma, and the released atoms move through the vacuum environment and deposit on a substrate to create a thin film [19]. Similar behavior has been reported by or Our result agrees well with [20][21],[22].

**Experimental part**

CdO-Ag Thin films on glass substrates have been deposited which use DC reactive magnetron sputtering technique. An evacuation of the deposition chamber was conducted in the first step at baseline pressure of $4 \times 10^{-2}$ mbar by using rotating mechanical pumps, and the second step was by the Turbo molecular that works directly to achieve the vacuum to $9 \times 10^{-4}$ torr. Oxygen and argon with high purity 99.995 gases They were being used as reactive gasses, and sputtering., with flow rate for argon 90 sccm and oxygen 10 sccm. Cadmium as a Metal target with a purity of 99.999 made by USA Torr International Inc, Diameter 50 mm, thickness 3 mm. And silver metal with purity 99.998, USA, Torr International Inc used as doping metal and shaped as chips with cylinder like with 2.5 mm diameter and height 2mm that pushed in the suitable hole made just fit in the target surface. The two faces of the target were being polished to look like a mirror to obtain higher conductivity for the target and High purity for the films deposited. Pressure has been reached at $8 \times 10^{-2}$ torr which is considered the pressure of deposition which is total oxygen and argon pressures. Target distance to substrate was quite 4 cm. The current of sputtering and voltage and voltage of sputtering have been kept throughout the process of deposition at 18 mA and 1300 V, consecutively. The CdO-Ag thin films have been prepared on substrates of glass at 150ºC with the use of electronic temperature controller, time of sputtering was kept 120 min for every single sample les. Figure (1) represents the devices and materials used in the experimental part.
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Fig. (1): (a) DC reactive magnetron sputter system, (b) plasma discharge of Oxygen gas, (c) film deposited on a glass substrate, (d) Cd target with 3 attached Ag chips, and (e) Ag chips

Deposition of CdO: Ag Thin Films procedures

1- Cadmium Target without any ads chips fixative in the pole of cathode through the holder in the technique of DC reactive magnetron sputtering. The temperature of the substrate has been set to 150°C.

2- Turn the vacuum system on that consist of two levels, first level the rotary vacuumed the chamber until 10⁻³ torr and in the second level the Turbo molecular that works directly to achieve the vacuum to 10⁻⁵ torr. The pressure inside the chamber monitoring by using Adixen ACC 2009 Pirani- Penning gauge.

2- As the pressure inside the chamber reaches to limit desired 9×10⁻⁴ torr, once Argon gas with a rate of Flow 90% sccm and Oxygen gas with a rate of flow 10 % sccm was admitted through the flow controller into the chamber to get a sputter pressure limit 8×10⁻² torr.

4- The applied DC sputter volt set to 1300 volts and the current on 18 mA,

5- As the sputtering processes countenance, watching the pressure counter, temperature, reflected power and chillier, very important to avoid any change in the sputter parameters.

6- Once we get the required sputter time 120 min the shutter has to be closed, turn the DC power and the vacuum system off, and venting the chamber.

Results and Discussions

Energy dispersive X-Ray spectroscopy (EDS) is performed during SEM analysis in order to determine the elemental composition of the film, Figure (1) Displays the cadmium and silver Ingredients in CdO:Ag films with different number of Ag chips As determined by the energy dispersive X-ray spectroscopy EDS by atomic percentage at.%. The findings are Ag contains in the films almost linearly increased with increasing of Ag chips [23]. The Ag contain in the CdO:Ag film of the target of cadmium with 1 bonded Ag chips is 0.4 at.%. It was increasing to 1.04 at. % when 2 Ag chips was bounded on the target of cadmium. Additionally increasing in the number of Ag chips to 3 the Ag concentration in CdO:Ag
composite films are increased further to 1.20 at.%. The indepth elementary compositional details relating the atomic percentage was displayed in table (1).

![Graph showing concentration of elements](image)

**Fig. (2): Silver, cadmium and Oxygen Concentration in the CdO: Ag DC Reactive Magnetron Sputtering Deposited Films**

**Table (1) Detailed information on the elementary composition regarding the atomic percentage was mentioned in**

| Element | 0 chips | 1 chip | 2 chips | 3 chips |
|---------|---------|--------|---------|---------|
| Cd      | 52.66   | 54.28  | 54.56   | 53.07   |
| Ag      | 0       | 0.4    | 1.04    | 1.20    |
| O       | 45.47   | 42.99  | 42.71   | 44.11   |
| Si      | 1.87    | 2.33   | 1.69    | 1.62    |

Figure (3, a, b, c and d) Shows Film Elementary Composition Which was identified by using energy dispersive X-Ray spectroscopy and results verify that all the deposited pure and silver doped CdO films consist of Cd spectrum $K_{\beta}$ = 3.1 k eV, silver spectrum $K_{\alpha}$ = 2.91 k eV and oxygen spectrum $K_{\alpha}$ = 1.05 k eV only, also we can see silica (substrate) spectrum $K_{\beta}$ = 2.8 k eV, it can be indicate two things from EDS, first one the film deposited contains no contamination, and the second one is CdO phase formation \[23\].
Figure (4) displays the X-Ray diffraction (XRD) pattern of undoped CdO NPs and CdO NPs doped with Ag NPs, at different concentration (0.4, 1.04 and 1.20) at%, which prepared by using DC reactive magnetron sputtering method and deposited on substrate of glass. It is noted from the figure that all prepared films have a polycrystalline reflection pattern, and the dominate reflection is (111) plane at the angle $2\theta=33.35^\circ$ and the reflection intensity was increased with the increase of the doping concentration (0.4, 1.04 and 1.20) at% of Ag NPs that implanted to the target surface of cadmium. The crystallite size $D$, was calculated from the Scherer equation (1) \[24\] which increased from 17.17 nm to 19.03 nm by increasing the doping from 0.4 at% to 1.2 at% due to the occupation of the ionic radius of Cd$^{+2}$ cation is 1.02 Å, that smaller than that of Ag$^{-2}$ cation 2.0 Å, leads to a full defect sites in the distance between Interplanar distanced As can be seen from Figure (5) and which represents a set of mathematical statistics of the Prevailing reflection (111), through the application of origin analysis graphing software. It is noted from the figures increasing of the intensity of the reflection, which is represented by the variable $(Y_o)$ and a decrease the full-width at half maximum (FWHM). Also we note the shifting of the reflection angle to less $2\theta$, which represented by the variable $X_C$.

The Figures (6- a, b and c) which represents both the crystalline size $(D)$, Micro strain $\varepsilon$ and the Dislocation density $(\delta)$, respectively, Were Computed using the equations (1), (2) and (3) respectively?

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

$$\varepsilon = \frac{\lambda}{4 \sin \theta} \quad (2)$$
\[ \delta = \frac{1}{D^2} \text{ (lines / m}^2\text{)} \] (3)

It noticed a decrease of micro strain (\(\varepsilon\)) as well as the Dislocation density (\(\delta\)), with the crystalline size increasing due to the reduction of crystal defects in the lattice. The results of crystalline size, Microstrain and dislocation density values are shown in Table (2).

**Fig. (4):** pattern of X-Ray diffraction of undoped CdO and CdO: Ag deposited by DC reactive sputtering

**Fig. (5):** Represents a set of mathematical statistics of the Prevailing reflection (a) 0 at\%, (b) 0.4 at\%, (c) 1.04 at\% and (d) 1.20 at\%.

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Fig. (6): structural properties of the prevailing reflection: (a) crystallite size, (b) microstrain, and (c) dislocation density.

Table (2): structural parameters for CdO doped Ag films prepared by reactive DC sputter.

| Ag concentration at% | hkl | 2θ $\Gamma$ | FWHM $\Gamma$ | Crystallite size D (nm) | Micro strain x10^-5 m | Dislocations density x10^-15 |
|----------------------|-----|-----------|---------------|------------------------|-----------------------|-----------------------------|
| 0                    | 111 | 33.35     | 0.438         | 17.17                  | 18.28                 | 33.90                       |
|                      | 200 | 37.8      |               |                        |                       |                             |
|                      | 220 | 54.80     |               |                        |                       |                             |
|                      | 311 | 64.05     |               |                        |                       |                             |
| 0.4                  | 111 | 33.35     | 0.417         | 18.03                  | 17.40                 | 30.73                       |
|                      | 200 | 37.8      |               |                        |                       |                             |
|                      | 220 | 54.80     |               |                        |                       |                             |
|                      | 311 | 64.05     |               |                        |                       |                             |
| 1.04                 | 111 | 33.35     | 0.415         | 18.14                  | 17.33                 | 30.37                       |
|                      | 200 | 37.8      |               |                        |                       |                             |
|                      | 220 | 54.80     |               |                        |                       |                             |
|                      | 311 | 64.05     |               |                        |                       |                             |
| 1.20                 | 111 | 33.35     | 0.39          | 19.30                  | 16.29                 | 26.82                       |
|                      | 200 | 37.8      |               |                        |                       |                             |
|                      | 220 | 54.80     |               |                        |                       |                             |
|                      | 311 | 64.05     |               |                        |                       |                             |
Microstructures characterizations of synthesized samples have been examined by scanning electron microscopy SEM and transmittance electron microscopy TEM. Figure (7) shows the SEM and TEM images of the CdO and Ag NPs films, deposited on a smooth and homogeneous distributed, and the crystallites were very fine. The size ofParticle had been increased with the addition of Ag NPs at different concentrations (1, 2 and 3) chips to the main target surface Cd metal. Figure (7-e),( 8-f),(9-g) and (10-h), which represents Transmission Electron Microscopy TEM shows that spherical shaped nanoparticles has been achieved with different diameters varies from (25-40) nm when the addition of Ag nanoparticles was 1.20 at.%.

Fig.( 7): SEM of undoped CdO films which deposited by using DC reactive magnetron sputtering, (a) 200 nm and (e) 50 nm
Fig.(8): SEM of one chip of silver doped cadmium films which deposited by using DC reactive magnetron sputtering (a) 200 nm and (c) 50 nm

Fig.(9): SEM of two chips of silver doped cadmium films which deposited by using DC reactive magnetron sputtering (a) 200 nm and (c) 50 nm
Fig.(10): SEM of three chips of silver doped cadmium films which deposited by using DC reactive magnetron sputtering (a) 200 nm and (c) 50 nm.

Conclusions

The success of the process of CdO:Ag Nano thin films by deformation of cadmium metal by silver metal through planting chips method through the use of the DC reactive sputtering method. The occurrence of a linear increase approximately in the silver material with an increase of one chips of silver for each time and the production of Nano thin films with a poly-crystalline diffraction pattern like spherical Nano structure and the diameter ranging from (25-40) nm.

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