[1]Jasim Mohamed Hussein,
[2]Hawraa Abd Al-Amerr Saleh,
[3]Mazin Hamed Hasan,
[4]Ibrahim Ramadhan Agool,
[5]Nadir Fadhil Habubi,
[6] Shaymaa A. Hussein,
[7]Sami Salman Chiad

Effect of Silver on Synthesis and Characterization on Co_3O_4 Thin Films Prepared by Spray Pyrolysis Method

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Abstract: - The Spray Pyrolysis process deposited nanostructured Cobalt oxide (Co3O4) and Co3O4: Ag thin sheets wads. XRD analysis shows the preferred orientation of the polycrystalline of Co3O4 and Co3O4: Ag films is (311). The grain size changed as the silver concentration increased, going from 15.51 nm to 17.44 nm. As silver (Ag) concentration in Co3O4 films increased, the cluster grain size reduced from 57.46 nm to 45.00 nm, confirmed by surface topography. The overall transmittance was more than 95 % in the visible region for 1% to 3% Silver (Ag) content. The absorption coefficient shows a decrement via the increment of Silver content in Co3O4 films, even though the energy gap has been significantly reduced from (2.52 to 2.43) eV as silver concentration increases, extinction coefficient and refractive index decreased with the silver.

Keywords: Co3O4, Ag, thin film, XRD, AFM.

I. INTRODUCTION

Thin films made of cobalt oxide have recently gained much study attention due to their potential for usage in various fields [1-3]. They can be employed as negative electrodes in lithium-ion batteries [4-6], anodic electrochromic materials in smart window systems [7-9] and other applications. Cobalt oxides are typically non-stoichiometric, with an excess of oxygen. This high oxygen content results in a p-type semiconducting behavior [10, 11]. The three crystalline forms of cobalt oxide are CoO, Co_3O_4, and Co_3O_4 [12-14]. Co_3O_4 is one of the most significant transition oxides, with great attention in numerous sectors [15-17]. Different methods like, CVD [18], RF magnetron sputtering [19], the sol-gel procedure [20], atomic layer deposition [21], the co-precipitation method [22] and chemical spray pyrolysis (CSP) [23-27], were used to create Co_3O_4 thin films. Spray pyrolysis, one of these deposition methods, has several benefits, including affordability, adaptability, convenience for broad

[1] Anbar Education Directorate Ministry of Education Anbar, Iraq
Jasim1988mojamad@gmail.com

[2]General Directorate of Babylon Education Ministry of Education Babylon, Iraq
hawraaalhassani77@gmail.com
[3]Dept. Physics College of Education for Pure science University of Anbar Anbar, Iraq
sc.mazin1al_qasy@uoanbar.edu.iq

[4]Bilad Alrafidain University College, Diyala, Iraq
dr.agool@bauc14.edu.iq

[5]Dept. Engineering of Refrigeration and Air Conditioning Technologies Alnukhba University College Baghdad, Iraq
n.fadhil@alnukhba.edu.iq

[6]Department of Medical Laboratory Techniques, Al-Manara College for Medical Science, Iraq.

[7]Dept. Physics College of Education, Mustansiriyah University Baghdad, Iraq
dr.sami@uomustansiriyah.edu.iq

deposition areas, and the potential to create thin films with nanostructures. In the current work, we have used the spray pyrolysis approach to study the impact of Ag on structural, morphological, and optical features of Co_3O_4 thin films.

II. EXPERIMENTAL

In the current study, CSP was employed to create Ag-doped Co_3O_4 thin films on a glass substrate, which were then coated on glass slide substrates. The German-provided 0.1 M of $CoCl_2$ was dissolved in 1:1 deionized water Wdion and ethanol to create the Co thin films. The doping agent was silver trichloride ($AgCl_3$), supplied by PubChem India. It was diluted in Wdion , and a few drops of HCl were added to the mixture to clarify. The following preparation requirements apply: 400 ºC for the substrate, space between spout and substrate was 30 cm. flux average 5 mL/min, spray rate was 9 sec followed by 90 sec to avoid cooling. Carrier gas was Nitrogen. XRD-obtained structural parameters were employed to study film surface. Transmittance was done using a UV-Vis NIR spectrophotometer.

III. RESULTS AND DISCUSSIONS

Figure (1) shows XRD patterns of thin films made of undoped Co_3O_4 and Co_3O_4: Ag. The positions of the Co_3O_4 film's XRD peaks are situated at angles 2 (36.84o), (54.57o), (54.57o), and (65.52o), which correspond to the planes (220), (311), (422) and (440), which were fitted with ICDD card No (42-1467). The peak positions of Co_3O_4: Ag showed a small shift, which may have been attributed to the lattice strain that was doping in Co_3O_4 possessed on the material. Regardless of the level of Silver doping, the (311) plane continued to grow preferentially in all films [28, 29].

The typical grain size, denoted by the letter D, was calculated using [30-32]:

$$D = \frac{0.9\lambda}{\beta cos\theta} \qquad (1)$$

Where λ is the wavelength of the X-rays, β and θ are (FWHM) and Bragg angle, respectively. The crystallite size of Co_3O_4 films was measured to be 15.51 nm. However, this value increased to 17.44 nm as the percentage of silver doping increased continuously up to 3%, because silver nanoparticles' ionic radius (1.15) is larger than that of cobalt oxide nanoparticles (0.58); they occupy vacated spaces, which is why this occurs. [33, 34].

The dislocation density (δ) is measured via Eq. (2) [35-37].

$$\delta = \frac{1}{D^2} \qquad (2)$$

The strain (Ɛ) is evaluated via Eq. (3) [38-40].

$$\varepsilon = \frac{\beta cos\theta}{4} \qquad (3)$$

δ and ε are decreasing from (41.56-32.87) nm, (22.35- 19.87) nm. It implies that δ may decrease due to the dopant atoms moving from the crystallite's interior to the grain boundary [41, 42]. Structural parameters $P_{st.}$ via silver content are offered in Table 1 and Fig. 2.

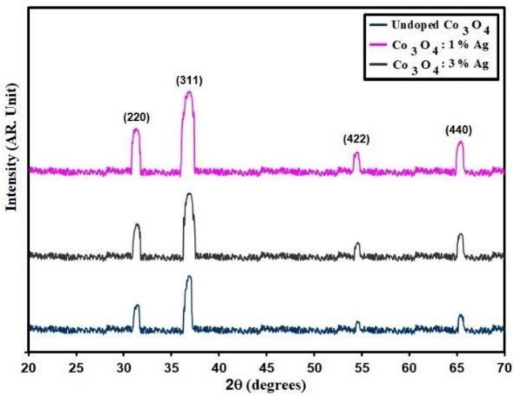

Fig.1. XRD-styles of intended films.

TABLE 1. *D*, E_g and P_{st} of grown films.

Samples	2 q (^{o})	(hkl) Plane	β (^{o})	E_g (eV)	D (nm)	dislocation density (δ) ($\times 10^{14}$) (lines/m^2)	strain (ε) ($\times 10^{-4}$)
Undoped Co_3O_4	36.84	311	0.54	2.52	15.51	41.56	22.35
Co_3O_4: 1% Ag	36.8	311	0.5	2.48	16.75	35.64	20.69
Co_3O_4: 3% Ag	36.76	311	0.48	2.43	17.44	32.87	19.87

Fig. (3) Doping ratios for AFM parameters (a1, b1, and c1), Fig. Displaying AFM pictures are (a2, b2, and c2). The average diameter (Dav) was recorded in the region of (57.46), (48.05) and (45.00) nm for the (Undoped Co_3O_4), (Co_3O_4:1% Ag), and Co_3O_4:3% Ag), respectively. R_{rms} value of 8.86 nm for Co_3O_4 thin films reduced to 6.12 nm with reduced silver content [43, 44]. In Fig., roughness parameters Ra were plotted against dopant concentration. (a3, b3, and c3), in that order. In Table (2), there is also a list of AFM parameters (P_{AFM}).

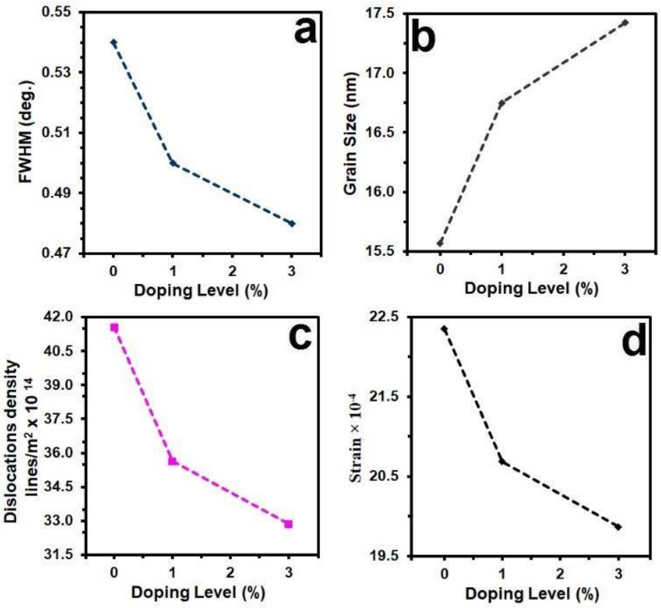

Fig.2. P_{st} of intended films.

TABLE 2. P_{AFM} of Ag doped and undoped Co_3O_4 thin films.

Samples	D_{av} nm	R_a (nm)	Rms (nm)
Undoped Co_3O_4	57.46	6.87	8.86
Co_3O_4: 1% Ag	48.05	6.38	6.46
Co_3O_4: 3% Ag	45	4.21	6.12

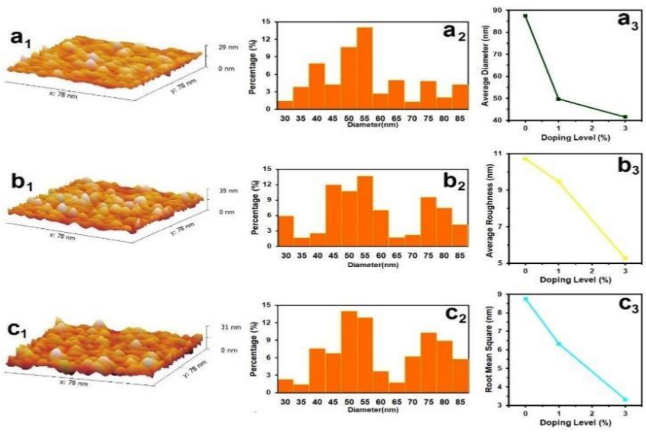

Fig. 3. PAFM of the grown films.

As illustrated in Fig. 4, a UV-visible spectrophotometer with a 300-900 nm wavelength range can record the transmittance (T) spectra. As seen in the image, the transmittance of undoped Co_3O_4 and Co_3O_4: Ag films decreased as the silver concentration increased [45, 46].

The absorption coefficient (α) is gained by Eq. 4 [47-49]:

$$\alpha = \ln (1/T)/d \qquad (4)$$

Where, d is film thickness. Figure (5) offers a graph of α plotted against the photon energy (hν), revealing that the α value exceeded 10^4 cm^{-1} for all films in the visible region. Additionally, an increase in absorbance was observed following the doping of the thin films with silver [50, 51].

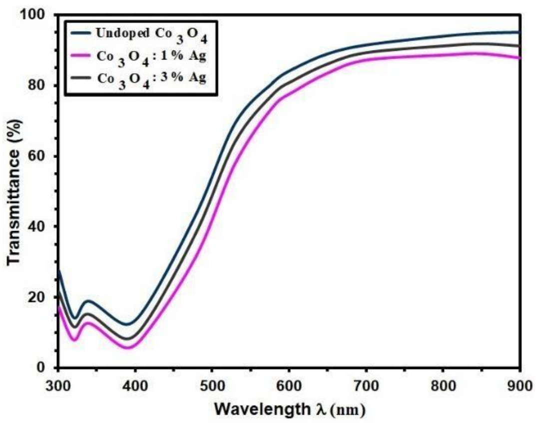

Fig. 4 T of grown films.

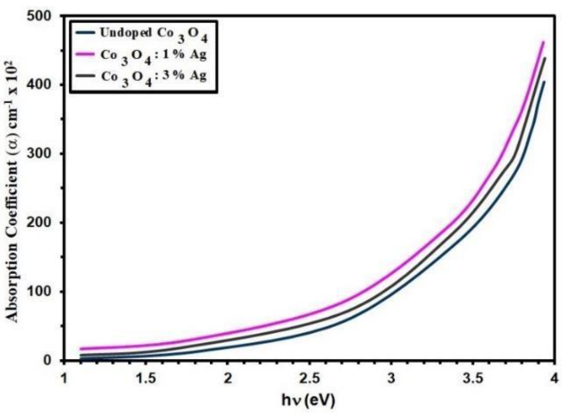

Fig. 5 α Vs hν for prepared films.

The band gap (E_g) of grown films for allowing direct transitions were calculated using the following equation [52-54]]:

$$(\alpha h\nu) = A(h\nu - E_g)^{\frac{1}{2}} \qquad (5)$$

Figure 6 illustrates Eg, where A is the constant. Undoped Co_3O_4, Co_3O_4: 1% Ag, and Co_3O_4: 3% Ag thin films have direct band gap energy values of around 2.52, 2.48, and 2.43 eV, respectively. This implies that adding silver reduces the energy gap by introducing doping energy levels. [55, 56]. Table (1) offers E_g values.

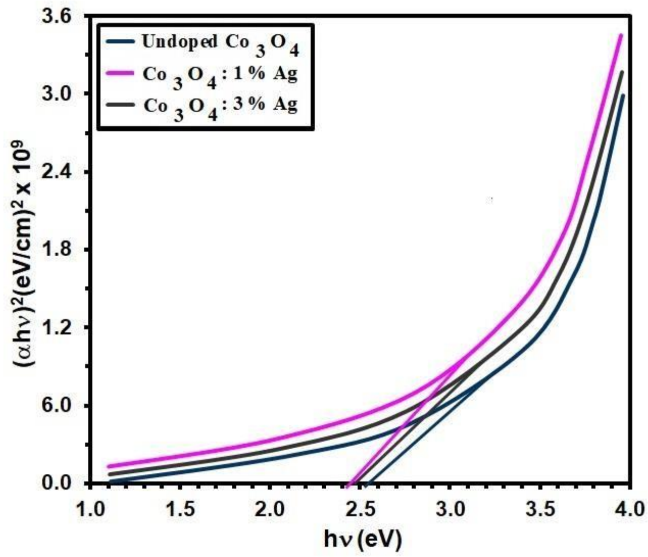

Fig. 6 E_g of grown films.

The relationship between the extinction coefficient, denoted by k, is plotted in Figure 7, and its value may be determined using the equation that is presented below [57-59]:

$$k = \frac{\alpha\lambda}{4\pi} \qquad (7)$$

Where, λ is the wavelength. k fell as the amount of silver in the mixture increased [60, 61].

The refractive index (*n*) is gained by employing Eq. 4 [62-64]:

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \qquad (8)$$

Where R is reflectance, Fig. 8 offers n via λ. n decreases with the increase of silver content [65, 66].

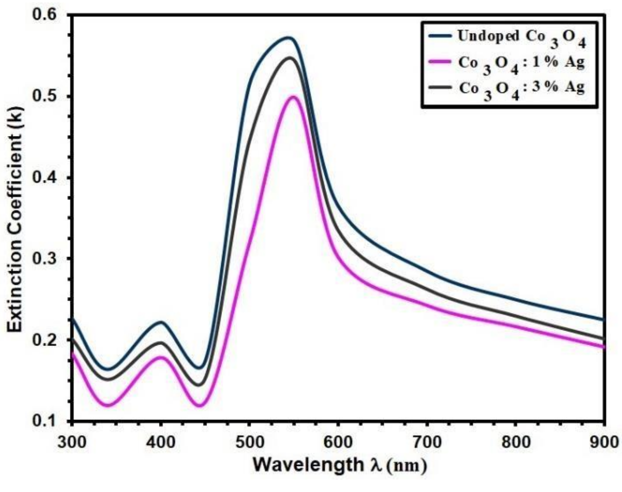

Fig. 7 k for the prepared films.

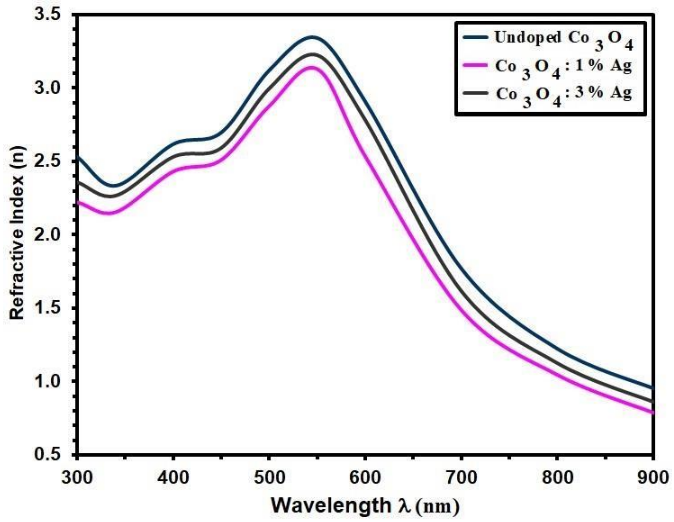

Fig. 8 n for the prepared films.

IV. CONCLUSION

Nanostructured cobalt oxide with 1% and 3% silver concentrations were deposited using the spray pyrolysis method (SPT). XRD analysis discovered the polycrystalline structure with the preferred orientation along (311) plane. The structural parameters represent dislocation density and strain; both decreased with silver content. The average particle size dropped to (57.46), (48.05), and (45.00) nm, respectively, while the average roughness decreased from 6.87 nm to 4.21 nm. Rrms values of grown films were (8.86, 6.46, and 6.129) nm. Atomic force microscopy (AFM) demonstrated homogeneous surfaces and regular atom distribution. In the visible spectrum, optical characteristics showed a high transmittance of over 95%, which declined with increasing silver concentration. The values of the absorption coefficients grow as the silver concentration increases, and the created films had values larger than 104 cm^{-1}. The optical energy gap decreased with the increment of Silver content from 2.52 to 2.43 eV, besides k and n decreased with the increment of silver content.

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