

Effect of Copper Doping on Physical Characteristic of Cobalt Oxide Nanostructured Thin Films Deposited for Gas Sensing

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Keywords: Co₃O₄, Cu, SPM, Structural Properties, Optical Band Gap, NO₂ Gas Sensing.

Abstract: (Co₃O₄: Cu) nanostructured films were deposited by the chemical spray pyrolysis (CSP) method. XRD analysis assure a polycrystalline cubic structure with peak at (220) plane. Improved crystalline quality was demonstrated by the crystallite size increasing from 16.96 nm for undoped films to 27.82 nm for 3% Cu-doped samples, and the dislocation density decreasing from 39.74×10^{12} to 30.41×10^{12} lines/m². Surface alteration was detected by AFM, and the average particle size decreased from 68.6 nm to 32.6 nm after doping. As Cu content rose, optical transmittance in the visible range dropped from 85.5% to 80.6%, whereas absorbance steadily increased. Due to defect-state formation within the band structure, the direct optical bandgap decreased from 2.50 eV to 2.40 eV. Strong light absorption was confirmed by the absorption coefficient exceeding 10^4 cm^{-1} . Because of structural alterations, the refractive index dropped as the Cu content increased. Copper doping greatly affected sensor response, as seen by gas-sensing experiments for NO₂ at 80°C. Sensitivity dropped from 53.62% for undoped Co₃O₄ to 29.43% for 3% Cu-doped films at 220 ppm, demonstrating decreased sensitivity at higher doping levels.

1 INTRODUCTION

Co₃O₄ thin films have garnered a lot of attention from researchers because of their potential uses in a variety of technical fields, such as use as negative electrode materials in lithium-ion batteries [1], [2], as anodic electrochromic layers for smart windows [3], [4], and as high-temperature solar selective absorbers [5], [6]. Co₃O₄ exhibits a spinel crystal structure characterized by intrinsic cation vacancies that enhance its chemical stability. This unique structural feature endows Co₃O₄ with exceptional properties that render it highly suitable for various electrochemical applications [8]-[10]. Several deposition techniques were used to create Co₃O₄, like RF magnetron sputtering [11], ALD [12], CBD [13], sol-gel processing [14], coprecipitation [15], PLD [16], CBD [17], and SPM [18]. Due to its potential for user-friendliness, low initial cost, and the capacity to deposit uniform thin films across a large surface with strict control

over composition and thickness, SPM has garnered a lot of attention [19]-[24]. The sensing applications of Undoped Co₃O₄ thin films are impacted by their modest sensitivity and weak electrical conductivity [25]-[27]. Copper incorporation in Co₃O₄ thin films drastically alters the structural, optical, and sensing properties [28]. In this work, Cu-doped Co₃O₄ nanostructured thin films were developed via CSP, and the effect of copper doping on their structural, optical, and sensing properties was studied.

2 EXPERIMENTAL

Co₃O₄ film: Co₃O₄ was synthesized through the CSP method. The membranes were created by decomposing a 0.1 M solution of (CoCl₂.6H₂O) in 100 ml of redistilled water. The volume fractions used for copper chloride were 1% and 3%. The

optimization parameters established in this study indicated that the substrate should be maintained at 450 °C. The solution prepared was sprayed onto glass bases. The spray rate was set at 0.2 ml per spray, with a sprayer nozzle height of 28 cm. Each spray lasted 8 seconds, and the interval between consecutive sprays was 2 minutes, while the nitrogen carrier gas was maintained at 105 Pa. The target thickness was 340 ± 25 nm. The structural properties of the deposited films were examined using XRD. Surface topography was investigated with an AFM. A dual-beam spectrophotometer from Shimadzu, Japan, was used to measure transmittance over the wavelength range of 380-900 nm. Studies of gas sensitivity were usually carried out in a cylinder laboratory chamber that sized 16 cm in height and 8 cm in radius.

3 RESULTS AND DISCUSSION

XRD patterns of the Co_3O_4 thin films are presented in Figure 1. According to the ICDD database (Card No. 42-146), the diffraction peaks noticed at 2θ values of 31.38° , 37.52° , 54.62° , and 65.57° linked to (220), (311), (422), and (440) crystallographic planes, respectively. These results indicate that the films display a predominantly hexagonal structure, with the (220) plane representing the preferred orientation.

Average crystallite size (D) was calculated from the diffraction peak's full width at half maximum (β), the X-ray wavelength (λ), and the Bragg angle (θ) using the Scherrer equation [29], [30].

D ranged from 16.96 to 27.82 nm, demonstrating a significant improvement in crystallinity; this could be linked to the effect of copper doping [14].

Dislocation density (δ), which is inversely proportional to the square of the crystallite size, was determined following [31].

δ of Co_3O_4 films decreases from 39.74×10^{14} lines/ m^2 in the undoped sample to 30.42×10^{14} lines/ m^2 in the film doped with 3% Cu. This drop implies fewer lattice defects, indicating that Cu incorporation improves crystalline quality and enhances the material's structural stability, which in turn can influence its electronic and optical properties [32].

The strain (ϵ), which quantifies lattice distortions within the crystallites, was estimated from the crystallite size and diffraction peak broadening [33]. ϵ decreases from 26.43×10^{-4} for undoped samples to 17.72×10^{-4} for films doped with 3% Cu, as shown in Table 1. This reduction in strain suggests that Cu incorporation alleviates lattice distortions, thereby improving crystallinity [34]. The structural parameters (S_{par}) comprising D , δ , and ϵ are listed in Table 1. Figure 2 depicts the variability of FWHM, D , δ , and ϵ versus Cu doping.

AFM was used to analyze surface topography of Co_3O_4 and Co_3O_4 films doped with 1% Cu. The images shown in Figure 3 disclose granular structures with surface scattering. Surface morphology roughness (R_a) and root mean square roughness (RMS) were investigated. As illustrated in Figure 3, Cu doping significantly influenced surface characteristics, resulting a changes in average particle size P_{av} , R_a , and RMS values, which were measured as (68.6, 61.8, 32.6) nm, (8.35, 6.52, 4.61) nm, and (7.65, 4.63, 3.27) nm, respectively [34]. Table 2 verifies that Cu incorporation reduces RMS, R_a and P_{av} of the films. These findings imply that Cu dopants promote particle growth and alter surface texture.

Figure 4 shows transmittance (T) spectra of Co_3O_4 films. In the Vis area, T ranges from 85.5% to 80.6%. A higher T is noted at longer wavelengths, showing a decrease in light absorption in NIR area, suggesting that films permit more efficient transmission [35].

Table 1: Structural parameters and optical band gap of undoped and Cu-doped Co_3O_4 thin films.

Sample	(hkl) Plane	2θ ($^\circ$)	FWHM ($^\circ$)	E_g (eV)	D (nm)	$\delta \times 10^{14}$ (lines/ m^2)	$\epsilon \times 10^{-4}$
Undoped Co_3O_4	220	31.38	0.74	2.50	16.96	39.74	26.43
Co_3O_4 :1% Cu	220	31.34	0.70	2.45	22.34	38.25	20.69
Co_3O_4 :3% Cu	220	31.31	0.66	2.40	27.82	30.41	17.72

Table 2: AFM surface parameters of undoped and Cu-doped Co_3O_4 thin films.

Sample	P_{av} nm	R_a (nm)	RMS (nm)
Co_3O_4 Undoped	68.6	8.35	7.65
Co_3O_4 :1% Cu	61.6	6.52	4.63
Co_3O_4 :3% Cu	32.6	4.61	3.27

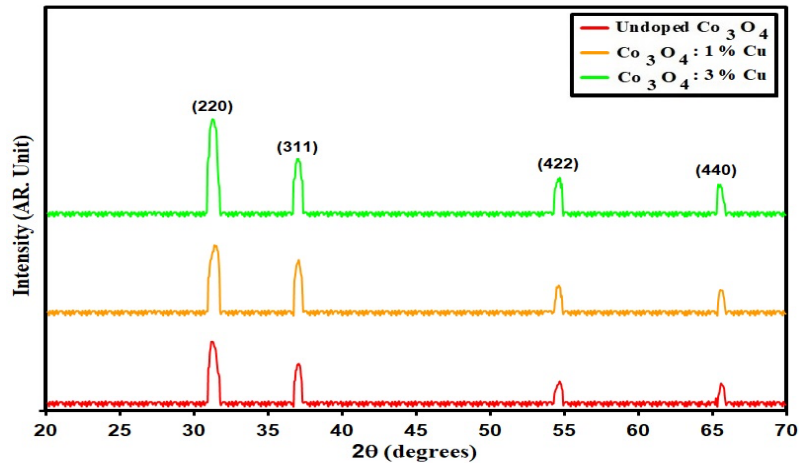


Figure 1: XRD patterns of undoped and Cu-doped Co_3O_4 thin films.

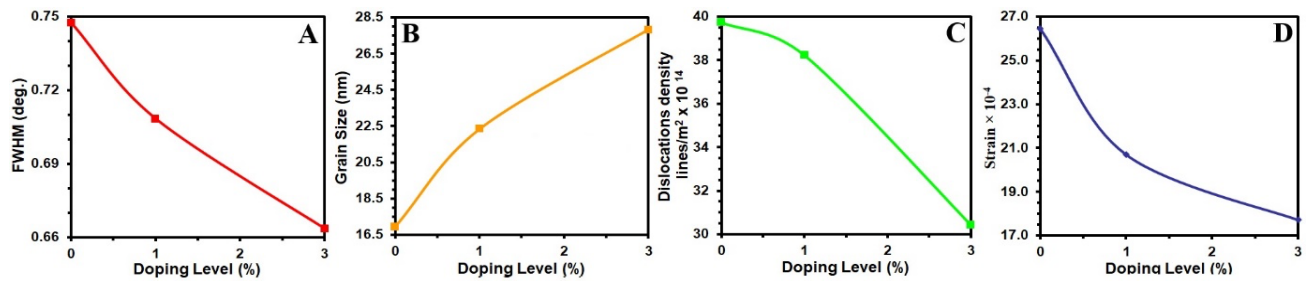


Figure 2: Variation of structural parameters (FWHM, crystallite size, dislocation density, and lattice strain) with Cu doping concentration.

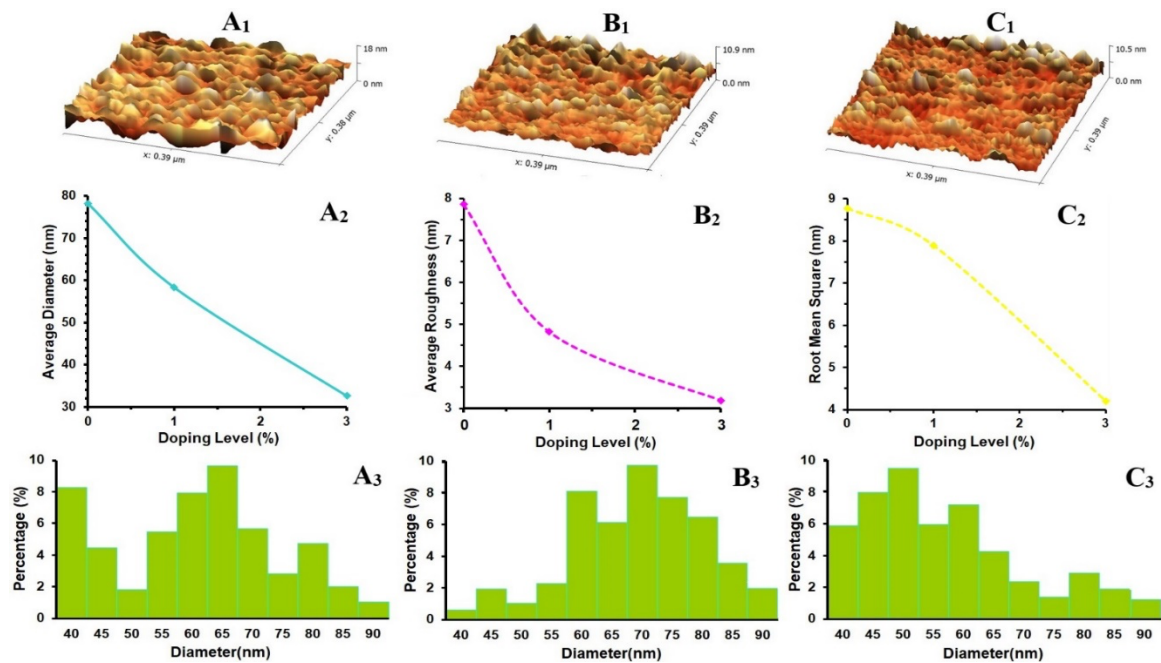


Figure 3: AFM surface morphology of undoped and Cu-doped Co_3O_4 thin films.

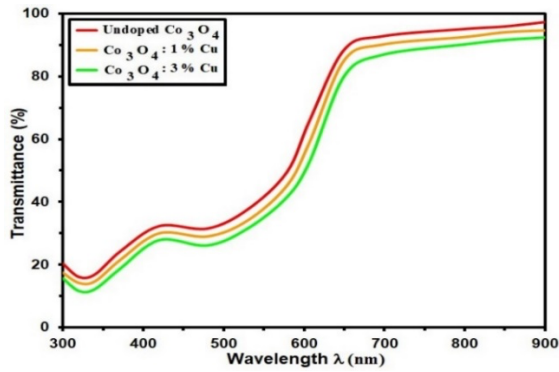


Figure 4: Optical transmittance spectra of undoped and Cu-doped Co_3O_4 thin films.

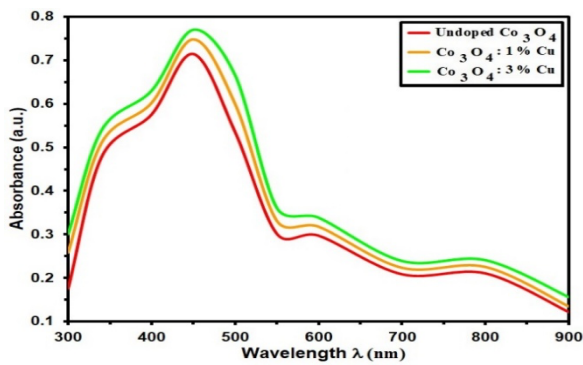


Figure 5: Optical absorbance spectra of undoped and Cu-doped Co_3O_4 thin films.

Absorbance (A) was evaluated from the transmittance (T), based on the intensity of transmitted light (I) and incident light (I_0) [36].

A is presented in Figure 5. The Co_3O_4 film shows the lowest A , while films doped with 1% and 3% Cu exhibit higher A . The increase in A is mostly ascribed to the incorporation of Cu ions, which introduce additional electronic states within the Co_3O_4 lattice [37].

The absorption coefficient (α) was obtained from the absorbance and film thickness (t) [38], [39].

Figure 6 shows that α initially rises sharply and then gradually decreases at higher energies. The high values of α ($\geq 10^{-4} \text{ cm}^{-1}$) indicate strong light absorption and efficient direct electronic transitions within Co_3O_4 films. The observed enhancement of α with Cu inclusion suggests improved photon absorption efficiency, which can be ascribed to the formation of new defect states [40], [41].

The direct bandgap (E_g) was determined from the Tauc relation between the absorption coefficient and photon energy, taking $n = 1/2$ for a direct allowed transition [42].

Figure 7 illustrates that E_g of Co_3O_4 films decreased with increasing Cu content. This reduction is ascribed to Cu-induced defect states, which introduce additional energy levels within E_g . Consequently, E_g of Co_3O_4 declined from 2.50 eV to 2.40 eV at the highest Cu doping level. The observed narrowing of E_g can be explained by the increased structural disorder and higher defect density induced by Cu incorporation, which alter the electronic structure and facilitate easier electronic transitions [43], [44].

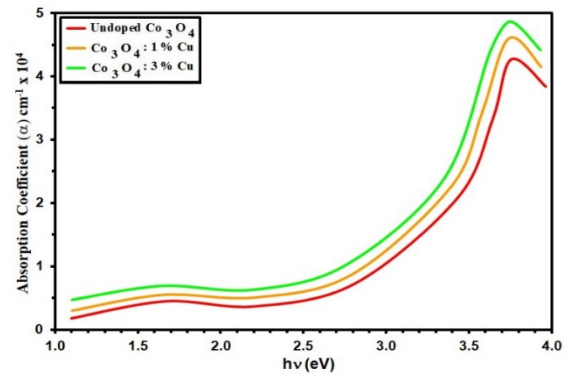


Figure 6: Absorption coefficient (α) as a function of photon energy ($h\nu$) for undoped and Cu-doped Co_3O_4 thin films.

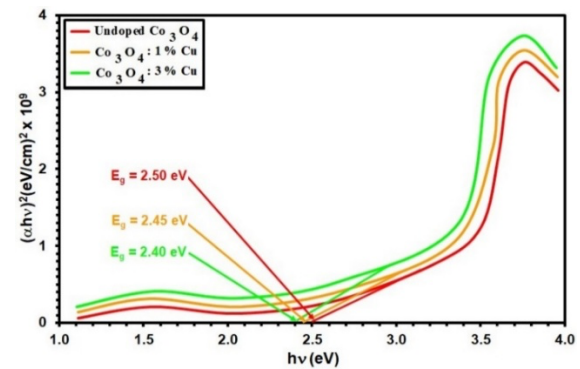


Figure 7: Tauc plots ($(\alpha h\nu)^2$ versus $h\nu$) of undoped and Cu-doped Co_3O_4 thin films.

The extinction coefficient (k) was calculated from the absorption coefficient (α) and wavelength (λ) [45].

As shown in Fig. 8, k tends to rise rapidly in the ultraviolet (UV) region before declining, even at photon energies ($h\nu$) below the fundamental absorption edge, which suggests that impurities or defect states in the film cause some absorption [46].

The refractive index (n) was computed from the reflectance (R) and extinction coefficient (k) [47].

Figure 9 displays n of Undoped Co_3O_4 films as a function of wavelength. All Co_3O_4 :Cu films have a refractive index that is directly correlated with wavelength. Additionally, as copper content increases, the refractive index decreases. This drop in (n) at higher doping concentrations can be accounted for by changes in electrical polarizability and structural alterations induced by copper incorporation, which affect the material's capacity to transmit light [48], [49].

At 80°C , the gas sensing properties were evaluated. The resistance (R) change when exposed to (NO_2) gas at a 200-ppm concentration is shown in Figure 10. Undoped Co_3O_4 films were found to have the lowest R , suggesting better adsorption of NO_2 molecules [50]. On the other hand, the Undoped Co_3O_4 films doped with 3% copper exhibited the highest R . This implies that copper doping increases (R) and alters the gas-sensing properties of Undoped Co_3O_4 films [51]-[53]. Sensitivity was then determined from the relative change in resistance between air and gas exposure [54], [55].

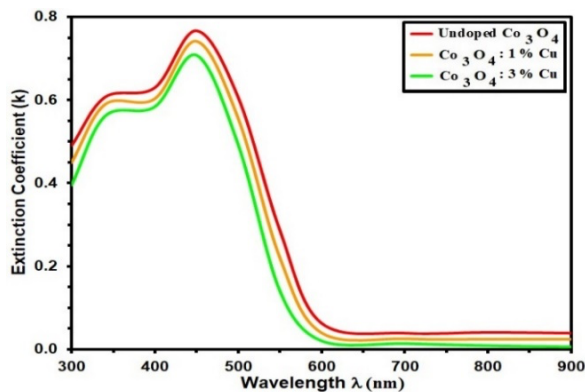


Figure 8: Extinction coefficient spectra of undoped and Cu-doped Co_3O_4 thin films.

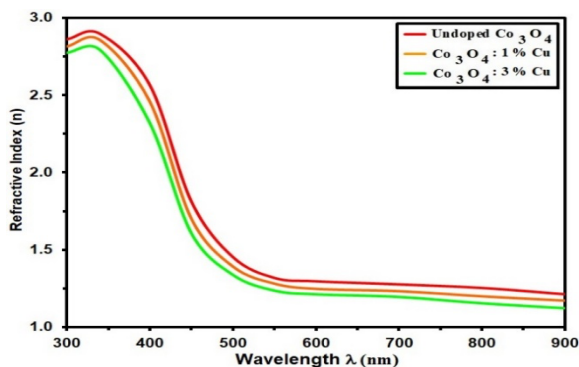


Figure 9: Refractive index spectra of undoped and Cu-doped Co_3O_4 thin films.

R_a represents the resistance in air and R_g represents the film sensor's resistance in the presence of gas. The sensitivity curves showing the effects of undoped Co_3O_4 , Co_3O_4 : 1% Cu, and Co_3O_4 : 3% Cu upon exposure to NO_2 gas are shown in Figure 11. The sensitivity is affected by charge carrier recombination between electrons and holes, which decreases with increasing copper doping concentration. In particular, the sensitivity decreases from 49.21% to 26.43% at 120 ppm, from 51.54% to 27.89% at 170 ppm, and from 53.62% to 29.43% at 220 ppm for undoped Co_3O_4 , Co_3O_4 : 1% Cu, and Co_3O_4 : 3% Cu. This drop in sensitivity indicates that higher copper concentrations make the sensor less sensitive to NO_2 [56], [57]. This effect is explained by structural changes, which affect the sensor's performance [58]-[60].

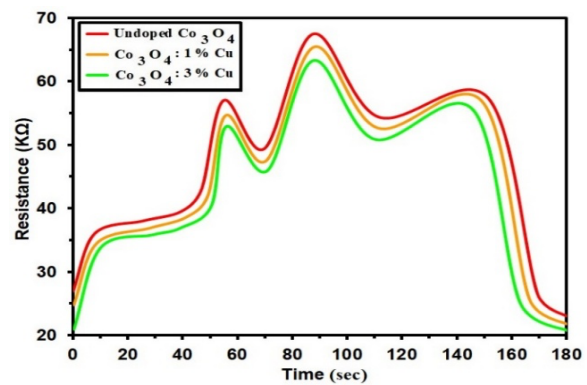


Figure 10: Resistance response of undoped and Cu-doped Co_3O_4 thin films under NO_2 gas exposure.

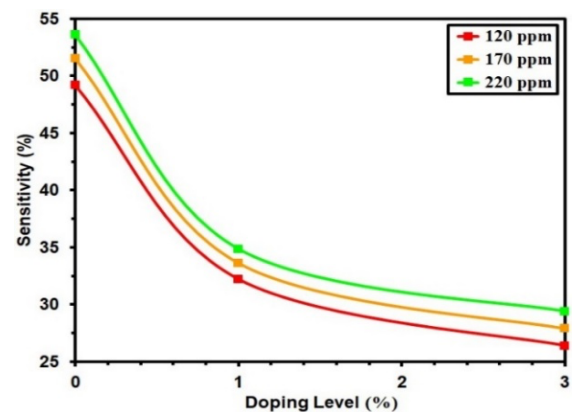


Figure 11: NO_2 gas sensitivity of undoped and Cu-doped Co_3O_4 thin films.

4 CONCLUSIONS

In this study, undoped and Cu-doped Co_3O_4 nanostructured thin films were successfully synthesized via SPM at 450°C . XRD analysis confirmed that all films are polycrystalline with a cubic structure and exhibit a strong peak along the (220) plane. D increased from 16.96 nm to 27.82 nm while reducing dislocation density and microstrain. AFM findings proved that average particle size and surface roughness were reduced. The transmittance decreased with increasing Cu concentration, while absorbance. The optical bandgap narrowed from 2.50 eV for Co_3O_4 to 2.40 eV for 3% Cu-doped films. Additionally, n decreased with higher Cu content. Gas-sensing tests for NO_2 at 80°C proved that sensitivity decreased from 53.62% to 29.43% at 220 ppm with increasing Cu doping.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial and institutional support provided by Mustansiriyah University (www.uomustansiriyah.edu.iq) for this research.

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