

Effect of Silver Doping on the Physical Properties of Zirconia Dioxide Nanostructures Thin Films

Athraa Mohammad Abdullah¹, Tariq Juma Mahmood¹, Murtada Kudhier Abbas Hassan²,
Karrar Noor Hussein³ and Taghreed Khalid Hameed⁴

¹Directorate of Education Baghdad Al-Karkh 2, Ministry of Education, 10001 Baghdad, Iraq

²Department of Scientific Affairs, Mustansiriyah University, 10052 Baghdad, Iraq

³Department of Radiology, Al-Manara College for Medical Sciences, 62001 Amarah, Iraq

⁴Department of Physics, College of Education, Mustansiriyah University, 10052 Baghdad, Iraq
taghreedkhalid@uomustansiriyah.edu.iq

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Abstract: Chemical spray pyrolysis (CSP) was employed to deposit ZrO₂:Ag thin films with good uniformity and adhesion. X-ray diffraction analysis confirmed the formation of a polycrystalline cubic phase, while Ag incorporation promoted grain growth and improved crystalline quality through the reduction of dislocation density and lattice strain. Atomic force microscopy (AFM) revealed a progressive decrease in surface roughness and particle size with increasing Ag content, indicating the development of a denser and more compact film morphology. Optical transmittance measurements showed moderate transparency in the range of 72.6-76.2%, with a slight decrease upon doping, although the films retained good transparency in the visible region. The optical band gap was found to narrow from 5.18 eV to 5.07 eV, which can be attributed to the introduction of localized defect states within the band structure. Gas-sensing measurements performed at 200 ppm NO₂ and an operating temperature of 130 °C demonstrated that undoped ZrO₂ films exhibited the highest sensitivity while Ag doping led to a gradual reduction in sensing performance.

1 INTRODUCTION

Zirconia thin films (ZrO₂) are classified as transition-metal oxides [1], [2] and exhibit attractive physical, optical, and chemical properties [3]-[6]. It has shown excellent hardness, very low electrical conductivity, high melting point, excellent thermodynamic stability [7], high refractive index, large dielectric constant, and a wide optical band gap [8]-[12]. It is thanks to these properties that ZrO₂ thin films have attracted tremendous interest as well as applications in other study areas like optical filtering [13], memory devices [14], and use as capacitors [15] in dynamic random-access memory [16]. Researchers have consequently looked into the creation of ZrO₂ thin films on a variety of substrates using a variety of techniques due to the vast range of essential applications. For example, pulsed laser deposition [17], the electron beam evaporation method [18], sputtering a ZrO₂ target using an argon-oxygen gas mixture at varied total gas pressures [19], sol-gel [20], RF sputtering deposition [21], and direct current magnetron sputtering technique [22].

Recently, metal element doping of zirconium dioxide thin films has been extensively investigated to improve their structural, optical, and functional properties [23], [24]. Among the diverse dopants, silver (Ag) has attracted much attention due to its remarkable electrical conductivity, catalytic properties, and strong affinity for oxygen species [25], [26]. The insertion of Ag into the ZrO₂ lattice can lead to modifications in the crystal structure, changes in the optical band gap, and an enhancement in surface activity that could be beneficial for sensing applications [27]. Ag-doped ZrO₂ thin films were created in this work using CSP) process. This study's primary goal is to examine how Ag doping affects the structural, optical, and sensing properties of ZrO₂ thin films.

2 EXPERIMENTAL

Thin ZrO₂:Ag films were deposited through the CSP technique. 0.1 M (ZrOCl₂.8H₂O) was dissolved in 100 milliliters of redistilled water to prepare the

solution. Doping with 2% and 4% Ag was applied. 400°C was maintained as the base temperature. A spray rate of 0.2 ml/spray, a 28 cm distance from the base to the sprayer, a 9-second spray duration, an interval between subsequent sprays of 1.5 minutes, and 105 Pa of air as the transport gas were considered suitable parameters. The gravimetric measurement indicated a film thickness of about 325 ± 25 nm. XRD measured the structural features. The surface morphology was characterized using atomic force microscopy (AFM) with an SPM-AA3000 scanning probe microscope (Angstrom Advanced Inc.). Transmittance was obtained using a double-beam spectrophotometer. The gas sensing performance was assessed by measuring the variation in electrical resistance within a custom-designed cylindrical chamber (9 cm radius and 19 cm height) under controlled H_2 gas exposure conditions.

3 RESULTS AND DISCUSSION

Figure 1 presents the XRD patterns of the synthesized films, confirming the formation of a well-defined crystalline structure. The diffraction peaks observed at 24.48° , 31.42° , 49.26° , and 63.22° were indexed to the (011), (111), (220), and (222) crystallographic planes, respectively. These diffraction peaks are consistent with the ICDD standard (File No. 37-1484), indicating the formation of a cubic phase.

The crystallite size was estimated using the Scherrer method, while the dislocation density and lattice strain were calculated according to the standard equations reported in the literature [28]–[32]. The calculated structural parameters are

summarized in Table 1. The results show that increasing the Ag concentration promotes crystallite growth, with the average crystallite size increasing from 17.26 to 32.10 nm. This behavior suggests that silver doping enhances atomic diffusion and facilitates crystallite coalescence during film growth, leading to the formation of larger crystallites [22].

In contrast, both the dislocation density and lattice strain gradually decreased as the Ag concentration increased. The dislocation density decreased from 39.34 to 24.38 , while the lattice strain decreased from 27.58 to 20.08 . These changes indicate an improvement in the crystalline quality of the films, as lower dislocation density and lattice strain are associated with fewer lattice defects and reduced internal stresses within the crystal structure [15], [16]. The variation of FWHM, crystallite size, dislocation density, and lattice strain with Ag concentration is presented in Figure 2.

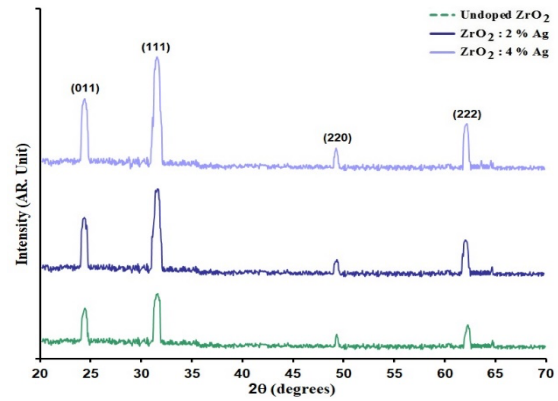


Figure 1: XRD patterns of undoped and Ag-doped ZrO_2 thin films.

Table 1: Structural parameters and optical band gap of undoped and Ag-doped ZrO_2 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 ZrO_2	100	31.42	0.48	5.18	17.26	39.34	27.58
ZrO_2 : 2% Ag	100	31.38	0.44	5.13	29.32	27.21	24.82
ZrO_2 : 4% Ag	100	31.33	0.40	5.08	32.10	24.38	20.08

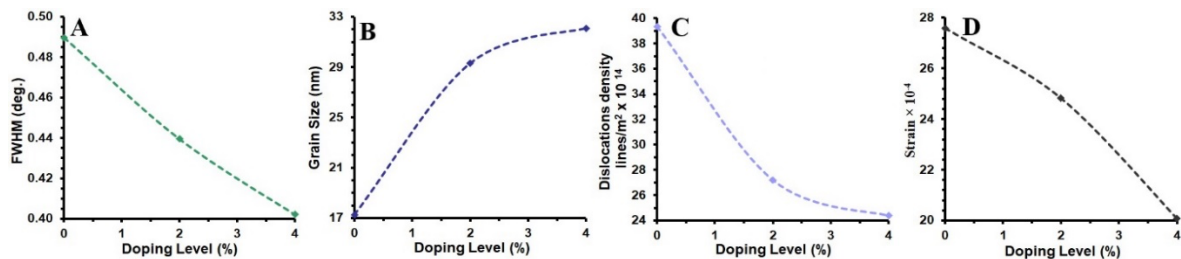


Figure 2: Structural parameters of undoped and Ag-doped ZrO_2 thin films: a) FWHM, b) crystallite size, c) dislocation density, and d) lattice strain as a function of Ag concentration.

Atomic force microscopy (AFM) was employed to investigate the surface morphology of pure and Ag-doped ZrO_2 thin films. The AFM images presented in Figure 3 reveal a homogeneous granular morphology with nanostructured grains uniformly distributed over the film surface. Surface characteristics were evaluated in terms of average particle size (P_{av}), average roughness (Ra), and root-mean-square roughness (RMS), and the corresponding values are summarized in Table 2.

Table 2: AFM parameters of ZrO_2 : Ag films.

Specimen	P_{av} (nm)	Ra (nm)	RMS (nm)
undoped ZrO_2	78.6	6.35	7.92
ZrO_2 : 2% Ag	54.1	4.16	6.16
ZrO_2 : 4% Ag	41.4	3.08	3.28

The results demonstrate that Ag incorporation significantly modifies the surface morphology of the films. The average particle size decreased from 78.6 nm for undoped ZrO_2 to 54.1 nm and 41.4 nm for the films doped with 2 and 4% Ag, respectively. Likewise, the average roughness (Ra) decreased from 6.35 to 4.16 and 3.08 nm, while the RMS roughness decreased from 7.92 to 6.16 and 3.28 nm. These results indicate that Ag doping suppresses grain

growth and promotes the formation of a smoother and more compact film surface, most likely by increasing the density of nucleation sites during film growth [33].

The optical transmittance spectra of the deposited films are shown in Figure 4. All samples exhibit relatively high transparency in the visible region, with transmittance values ranging from approximately 72.6% to 76.2%. The transmittance gradually increases with wavelength, reflecting reduced optical absorption at lower photon energies. This behavior is attributed to the insufficient photon energy required to excite electrons from the valence band to the conduction band or to defect-related energy levels [34].

The absorbance spectra shown in Figure 5 were calculated from the measured transmittance using the standard optical relationship [35]. The undoped ZrO_2 film exhibits the lowest absorbance, whereas the absorbance increases progressively with increasing Ag concentration. This enhancement is attributed to the incorporation of Ag into the ZrO_2 lattice, which introduces additional impurity and defect states within the band gap, thereby increasing the probability of electronic transitions and photon absorption [36], [37].

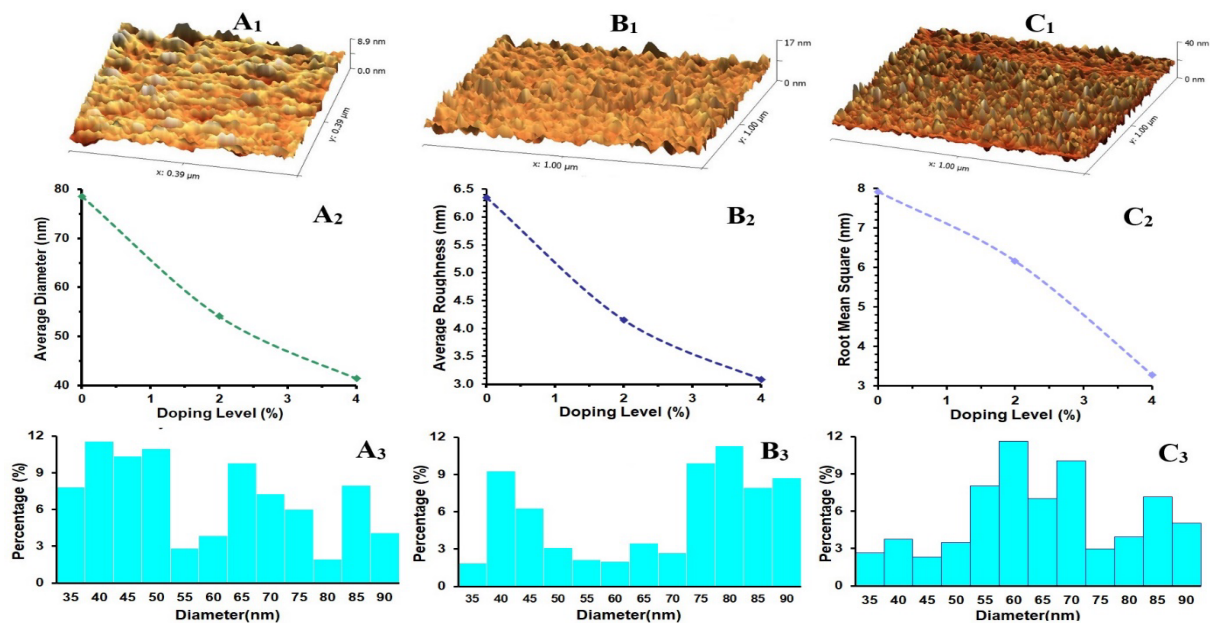


Figure 3: AFM surface morphology of undoped and Ag-doped ZrO_2 thin films.

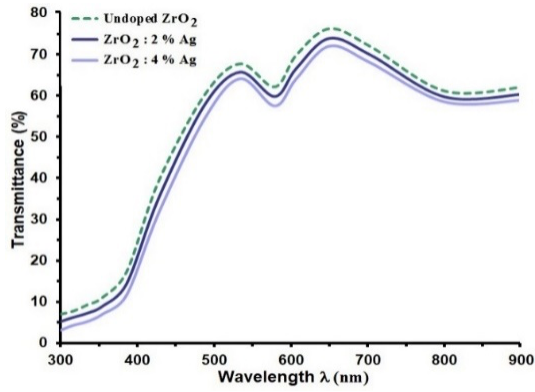


Figure 4: Optical transmittance spectra of undoped and Ag-doped ZrO₂ thin films.

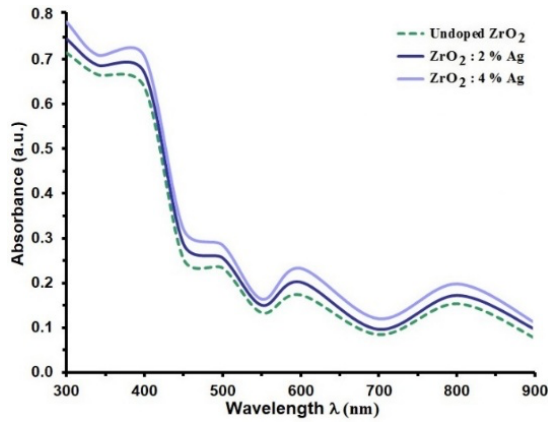


Figure 5: Optical absorbance spectra of undoped and Ag-doped ZrO₂ thin films.

The absorption coefficient was calculated from the absorbance spectra using the conventional relationship for thin films [38]. As shown in Figure 6, the absorption coefficient increases with Ag concentration, particularly near the absorption edge, indicating enhanced optical absorption. The obtained values exceed 10^4 cm^{-1} , confirming strong optical absorption and the predominance of direct electronic transitions. This enhancement is attributed to Ag-induced modifications in the electronic structure, which introduce additional localized states that facilitate photon absorption and electronic excitation [39], [40].

The optical band gap was determined using the Tauc method for direct electronic transitions [41]. Figure 7 shows that the optical band gap decreases progressively with increasing Ag concentration, from 5.18 eV for undoped ZrO₂ to approximately 5.07 eV for the film containing the highest Ag content. This reduction is attributed to the formation of impurity and defect states associated with Ag incorporation

into the ZrO₂ lattice. These localized energy levels narrow the effective band gap by facilitating electronic transitions at lower photon energies. In addition, Ag doping increases structural disorder, resulting in band-tail formation and a slight reduction in the optical band gap [42], [43].

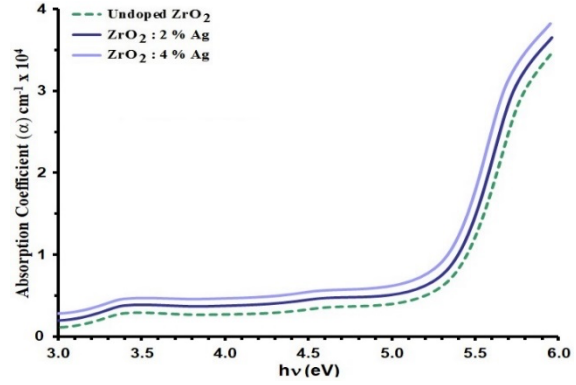


Figure 6: Absorption coefficient as a function of photon energy for undoped and Ag-doped ZrO₂ thin films.

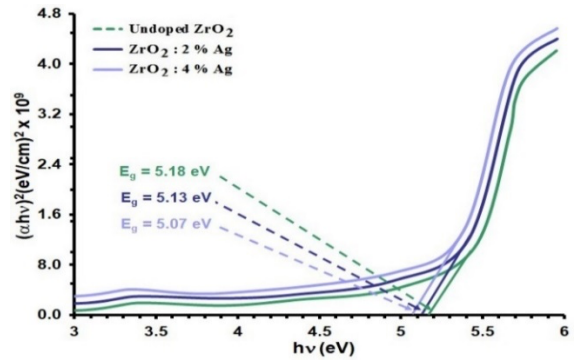


Figure 7. Tauc plots for determining the direct optical band gap of undoped and Ag-doped ZrO₂ thin films.

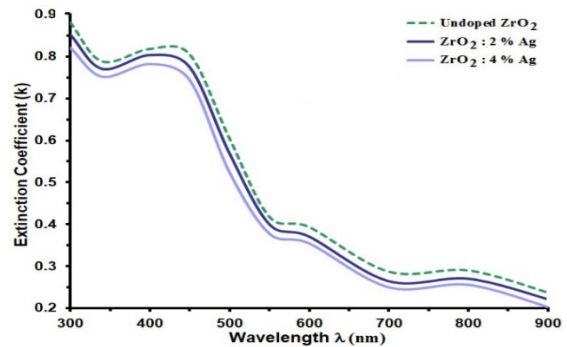


Figure 8: Extinction coefficient as a function of wavelength for undoped and Ag-doped ZrO₂ thin films.

The extinction coefficient was calculated from the optical absorption data using the standard relationship [44]. As illustrated in Figure 8, the extinction coefficient increases rapidly in the ultraviolet region and gradually decreases with increasing wavelength. The nonzero values observed below the fundamental absorption edge indicate the presence of sub-band-gap absorption, which is commonly associated with structural defects, impurity levels, and localized electronic states introduced during film growth and Ag incorporation [45].

The refractive index was determined from the optical reflectance data using the conventional model described in the literature [46,47]. Figure 9 shows that the refractive index exhibits normal optical dispersion, increasing gradually with wavelength for all samples. However, the refractive index decreases with increasing Ag concentration, indicating that Ag incorporation modifies the electronic polarizability and microstructure of the ZrO_2 films, thereby reducing their optical density [48], [49].

The gas-sensing performance of the films was evaluated at an operating temperature of 130°C using 200 ppm NO_2 gas. The resistance responses presented in Figure 10 show that the undoped ZrO_2 film exhibits the lowest electrical resistance, indicating more efficient interaction with NO_2 molecules and higher charge-carrier mobility. In contrast, the film containing 4% Ag exhibits the highest resistance, suggesting that Ag incorporation modifies the electronic structure and limits charge transport during gas adsorption [50], [51].

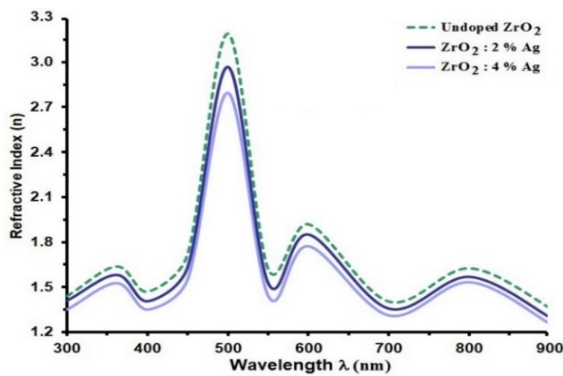


Figure 9: Refractive index as a function of wavelength for undoped and Ag-doped ZrO_2 thin films.

The sensor sensitivity was calculated using the standard resistance-based expression [52]. Figure 11 demonstrates that the sensitivity decreases with increasing Ag concentration over the investigated NO_2 concentration range. At 110, 160, and 210 ppm

NO_2 , the sensitivity decreases from 31.16% for undoped ZrO_2 to 6.76%, 32.29% to 7.95%, and 34.18% to 9.08%, respectively, for the films containing 2% and 4% Ag. This behavior indicates that excessive Ag incorporation reduces the gas-sensing response, most likely because dopant-induced structural and electronic modifications suppress charge-carrier transport and decrease the number of active adsorption sites available for NO_2 molecules [53]–[58].

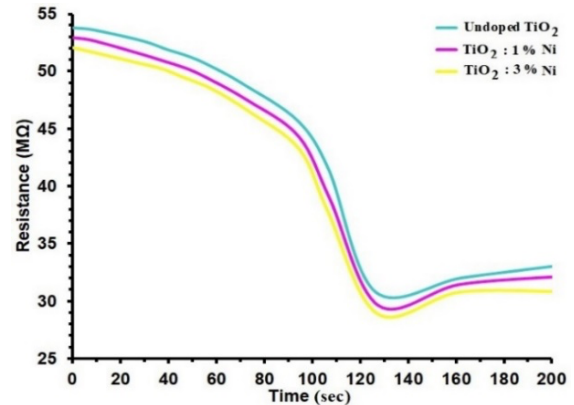


Figure 10: Dynamic resistance response of undoped and Ag-doped ZrO_2 thin films toward 200 ppm NO_2 at an operating temperature of 130°C .

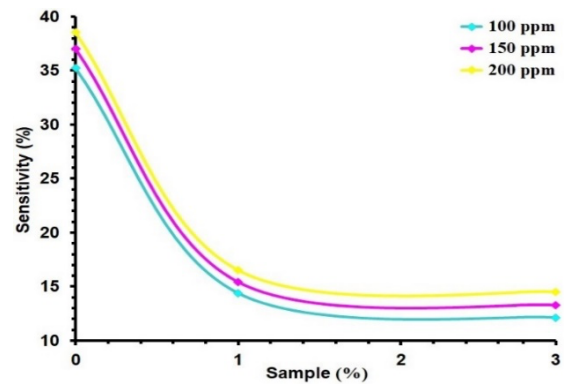


Figure 11: NO_2 gas sensitivity of undoped and Ag-doped ZrO_2 thin films at different gas concentrations.

4 CONCLUSIONS

In summary, Ag-doped ZrO_2 thin films were successfully deposited using the chemical spray pyrolysis (CSP) technique, demonstrating good reproducibility and uniform coverage. Structural characterization confirmed that the incorporation of Ag ions promotes crystallite growth, improves the film matrix. Additionally, surface morphology

analysis revealed a smoother and more homogeneous surface with increasing Ag concentration, accompanied by a noticeable reduction in average particle size (from AFM) and surface roughness, even though the average crystallite (grain) size determined from XRD increased with Ag content. Optical investigations indicated that all films maintained high transparency in the visible region, while the optical band gap decreased systematically from 5.18 eV to 5.08 eV with Ag doping, likely due to the introduction of localized energy states within the band structure. Despite these improvements, gas-sensing measurements toward NO₂ demonstrated that undoped ZrO₂ films exhibited the highest sensitivity, whereas Ag incorporation led to a gradual decline in sensing performance. This reduction may be attributed to altered charge transport mechanisms and reduced active adsorption sites. Therefore, optimizing dopant concentration is essential for balancing material properties and achieving application-specific performance.

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