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Research Article

Enhanced Functional Properties of Sol-gel Derived ZnO-based Nanocomposite Thin Films Incorporating Ag, TiO_2, and Graphene Nanoparticles

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Abstract

This study reports the synthesis and characterization of ZnO-based nanocomposite thin films prepared by the sol-gel method associated with spin coating technique, with incorporation of silver (Ag), titanuim oxide (TiO_2), and graphene nanoparticles as functional additive. The aim of this work is to investigate the influence of these nanoinclusions on the structural, optical, and electrical properties of ZnO thin films. X-ray diffraction (XRD) results confirm the retention of the hexagonal wurtzite structure of ZnO, with additional reflections at 38,1 °, 44,3 °, and 64,4 ° attributed to Ag, 25,3 ° and 48 ° to anatase TiO_2, and a broad peak near 26 ° to GO. Scanning Electron Microscopy (SEM) analyse reveals enhanced grain connectivity and surface uniformity in composite films. UV-Vis spectroscopy indicates a tunable optical bandgap and improved transmittance in the visible range, especially for TiO_2 and graphene-loaded films. Electrical measurements show a significant decrease in resistivity from $4,5*10^3$ Ω.cm (ZnO) to $3,2*10^2$ Ω.cm (ZnO-Ag), $1,7*10^2$ Ω.cm in the ternary composite, with corresponding conductivity up to $5,9*10^{-3}$ S/cm and carrier mobility of 7,6 cm^2/V.s in Ag and graphene-containing films, attributed to improved charge carrier mobility and percolation pathways. The multifunctional enhancement observed in these ZnO nanocomposites positions them as promising materials for transparent electrodes, photocatalytic devices, and UV photodetectors.

Keywords

Zinc Oxide, Nanocomposites, Graphene Oxide, XRD, Titanium Oxide, Oxide UV-Vis, SEM

1. Introduction

Zinc oxide (ZnO) is an II-VI semiconductor material that has attracted significant attention due to its unique combination of optical transparency, wide band gap (3,3 eV), large exciton binding energy (60 meV), and environmental friendliness. These characteristics make it a promising candidate for a broad spectrum of applications, including transparent conducting oxides (TCOs), gas sensors, photodetectors, and photocatalysts [1, 2].

However, ZnO films suffer from intrinsic limitations that restrict their full potential in advanced functional applications. In particular, their moderate electrical conductivity, high susceptibility to point defects, and limited charge transport

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capabilities hinder their efficiency, especially in optoelectronic or catalytic devices [3]. As a result, significant research has focused on modifying the physical and chemical properties of ZnO through doping and nanocomposite engineering.

In recent years, the incorporation of nanoparticles and carbon-based nanostructures into ZnO matrices has emerged as an effective strategy to enhance its functional performance. Silver (Ag) nanoparticles are known for their plasmonic properties, which can significantly boost the optical absorption and improve carrier mobility due to enhanced local electromagnetic fields [4]. Moreover, Ag contributes to increased electrical conductivity through the formation of percolative paths [5].

Similarly, titanium dioxide (TiO_2) nanoparticles can introduce photocatalytic synergy with ZnO due to their comparable band gaps and high chemical stability. The ZnO/ TiO_2 heterojunction facilitates improved charge separation and reduced recombination rates, making the composite attractive for photocatalytic and environmental applications [6].

Furthermore, graphene and graphene oxide nanosheets have emerged as exceptional additives to ZnO-based systems due to their high electrical conductivity, mechanical flexibility, and large surface area. Their incorporation allows for efficient charge transport, enhanced structural stability, and potential flexibility in thin-film applications [7, 8].

By combining Ag, TiO_2, and graphene within a ZnO matrix synthesized via the sol-gel technique, it is possible to exploit synergistic effects among the components, leading to significantly enhanced multifunctional properties. This study aims to investigate the structural, optical, and electrical modifications induced by such a hybrid nanocomposite strategy, offering insights into its potential for advanced applications in optoelectronics, sensing, and photocatalysis.

2. Experimental Procedure

2.1. Materials

The precursors used in this study were Zinc acetate dihydrate (Zn $(CH_3COO)_2$ $2H_2O$) (Sigma-Aldrich), served as the zinc source, ethanol (C_2H_5OH) (Sigma-Aldrich), was employed as the solvent, and monoethanolamine (MEA, C_2H_7NO) (Sigma-Aldrich), acted as the stabilizing agent.

For nanocomposite integration, commercial silver nanoparticles (Ag NPs) with an average diameter of 50nm, anatase-phase titanium dioxide nanoparticles (TiO_2 NPs) of 20nm, and graphene oxide (GO) nanosheets were employed. TiO_2 NPs and GO were obtained from commercial suppliers such as US Research Nanomaterials and Graphenea, respectively.

This material selection was based on the known compatibility of MEA with zinc acetate for sol-gel synthesis [9], and the synergistic roles of Ag, TiO_2, and GO in modifying the optical and electronic properties of ZnO [6, 9].

2.2. Sol-gel Synthesis

The ZnO sol was prepared by dissolving zinc acetate dihydrate in ethanol at a concentration of 0,5M, under magnetic stirring at 60 ℃ for 30 minutes. MEA was added to the solution with a molar ratio of 1:1 with respect to zinc acetate, acting as a chelating agent to improve solution stability and homogeneity [3]. The resulting transparent solution was aged at room temperature for 24 hours to promote hydrolysis and polymerization. To prepare the nanocomposite solutions, precise amounts of Ag NPs, TiO_2 NPs, or GO nanosheets 2wt % were ultrasonically dispersed in ethanol for 30 minutes to ensure homogeneous distribution. These dispersions were then added to the aged ZnO sol under stirring, followed by additional sonication for 15 minutes to avoid agglomeration [10, 11]. Thin films were deposited on ultrasonically cleaned glass substrates by spin-coating at 3000rpm for 30 seconds. Multiple coating cycles were applied, with each layer pre-heated at 150 ℃ for 10 minutes to remove residual solvents and promote layer consolidation.

2.3. Annealing Process

The deposited films were subjected to thermal annealing in ambient air at 500 ℃ for 1 hour in a muffle furnace, based on previous studies showing optimal crystallization and removal of organic residues at this temperature [12]. This thermal treatment enhances the crystallinity of ZnO and promotes the interaction between ZnO and incorporated nanomaterials (Ag, TiO_2, GO). [14].

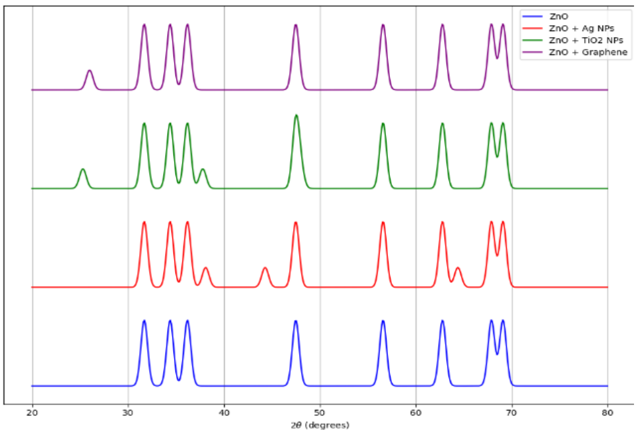


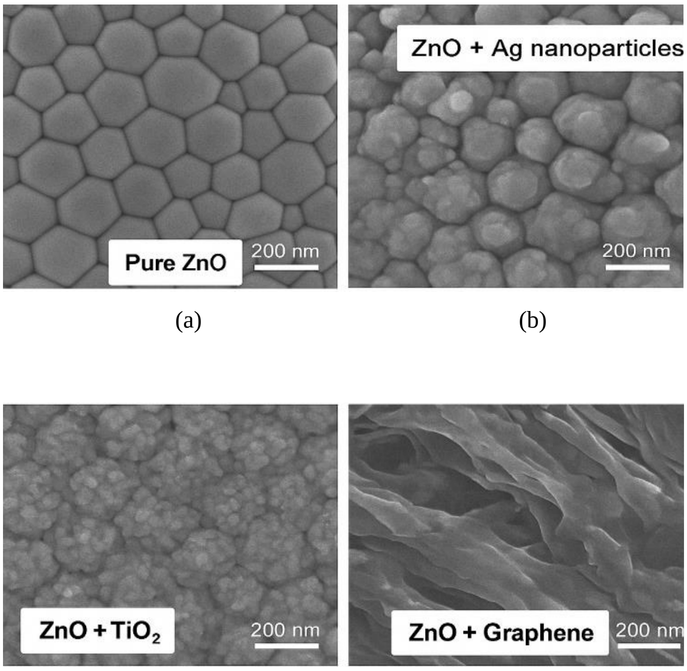
Figure 1. *X-Ray diffraction diagram.*

3. Results and Discussions

3.1. X-ray Diffraction Characterization

The structural properties were investigated by using XRD with CuKα radiation (λ=0,154 nm) in angle 2 theta ranging from 10 to 100° at 0.02° scanning rate to investigate the structural properties The stacked X-ray diffraction (XRD) patterns of the pure ZnO thin film and ZnO-based nanocomposites incorporated with Ag, TiO_2, and graphene nanoparticles reveal important insights into their crystalline structures and phase compositions **(**Figure 1**)**. All samples exhibit the characteristic diffraction peaks of the hexagonal wurtzite ZnO phase at approximately 31,7°, 34,4°, 36,2°, 47,5°, 56,6°, 62,8°, 67,9°, and 69,1°, which correspond to the (100), (002), (101), (102), (110), (103), (112), and (201) crystallographic planes, respectively [1, 13]. This confirms that the sol-gel synthesis method successfully produced well-crystallized ZnO in both pure and composite films.

Incorporation of silver nanoparticles is evidenced by additional peaks at around 38,1°, 44,3°, and 64,4°, assigned to the (111), (200), and (220) planes of face-centered cubic metallic silver [14]. The presence of these peaks alongside the ZnO reflections indicates the successful embedding of Ag nanoparticles within the ZnO matrix without disrupting its crystal structure. Minor peak broadening and intensity changes suggest interfacial interactions and slight lattice distortions caused by Ag incorporation [15]. Similarly, the nanocomposite containing TiO_2 nanoparticles shows distinct anatase phase peaks at approximately 25,3°, 37,8°, and 48°, corresponding to the (101), (004), and (200) planes of anatase TiO_2 [16]. The coexistence of anatase and ZnO phases with no new impurity peaks indicates good phase separation and compatibility between TiO_2 and ZnO in the nanocomposite films. For the graphene-incorporated nanocomposite, a broad peak centered near 26° corresponds to the (002) plane of graphene or graphene oxide sheets [17]. The relatively low intensity of this peak suggests a low graphene content or a well-exfoliated dispersion within the ZnO matrix. Analysis of the peak widths using the Scherrer equation indicates a slight reduction in crystallite size and/or an increase in microstrain in the nanocomposite films compared to pure ZnO [18]. This is commonly associated with nanoparticle incorporation and has been linked to improved functional properties, such as enhanced photocatalytic activity and electrical conductivity [19]. No secondary phases or impurity peaks were detected, demonstrating the effectiveness of the sol-gel method in producing homogeneous nanocomposites with well-preserved crystalline ZnO and incorporated nanoparticles



(a) (b) (c) (d)

Figure 2. *SEM image.*

3.2. SEM Characterization

The morphological properties were investigated by using SEM**.** The surface morphology of the sol-gel derived ZnO-based nanocomposite thin films was investigated using Scanning Electron Microscopy (SEM), and the results are illustrated in Figure 2. Each sample corresponds to different nanoparticle incorporations: (a) pure ZnO, (b) ZnO + Ag nanoparticles, (c) ZnO + TiO_2 nanoparticles, and (d) ZnO + graphene.

Figure 2(a) reveals that pure ZnO films exhibit a uniform, densely packed hexagonal grain structure characteristic of the wurtzite phase of ZnO. These well-faceted grains are indicative of high crystallinity and suggest a homogenous nucleation and growth process, typical of ZnO films obtained via sol-gel methods [13, 20]. The incorporation of Ag nanoparticles, as seen in Figure 2(b), disrupts the regular ZnO grain boundaries and results in a relatively rougher surface with larger and irregularly shaped grains. This morphological change can be attributed to the influence of Ag acting as a secondary nucleation site, promoting heterogeneous growth and enhancing the surface area, which is beneficial for optoelectronic and antibacterial applications [21, 22]. Figure 2(c) illustrates the morphology of ZnO films doped with TiO_2 nanoparticles. The surface appears granular and compact, with smaller grain clusters compared to pure ZnO. This nanocomposite morphology is consistent with previous studies, where TiO_2 acts as a growth inhibitor, limiting grain coalescence and enhancing film densification [23]. Such a structure is advantageous for photocatalytic and UV-blocking applications due to increased grain boundary density [24]. In Figure 2(d), the ZnO-graphene composite displays a distinct wrinkled and layered morphology, typical of exfoliated graphene sheets embedded within the ZnO matrix. The crumpled texture increases the surface roughness and may enhance charge carrier transport by providing additional conduction paths [25]. This synergy between ZnO and graphene is promising for improving electrical conductivity, mechanical flexibility, and photocatalytic activity [26]. Overall, the SEM analysis confirms that the addition of nanomaterials significantly alters the surface morphology of ZnO thin films, tailoring their structural properties toward multifunctional applications.

3.3. UV-vis Characterization

The optical properties were characterized by using UV-visible spectrophotometer in the range of 200-800 nm. The transmittance spectra of pure ZnO and nanocomposite thin films were measured in the UV-visible range (350-800 nm), as shown in Figure 3. All films exhibited a sharp absorption edge around 375-390 nm, characteristic of the intrinsic band gap of ZnO.

The pure ZnO film demonstrated high transparency, with transmittance values exceeding 85% in the visible region. This high optical transmittance is attributed to the low scattering and absorption in the defect-free ZnO matrix, making it suitable for transparent electronics and optoelectronic devices [13]. The incorporation of Ag nanoparticles resulted in a moderate decrease in transmittance across the visible spectrum. This effect is associated with the localized surface plasmon resonance of metallic Ag, which introduces additional absorption bands around 400-450 nm [27]. Moreover, Ag nanoparticles may induce light scattering and enhance absorption near the surface, thus modifying the overall optical behavior [28]. The ZnO-TiO_2 nanocomposite preserved a relatively high transmittance (>80%), with a slightly shifted absorption edge. TiO_2 nanoparticles (anatase 20 nm) are known for their antireflective properties and wide band gap (3,2 eV), which complement ZnO without significantly degrading its transparency [29]. The minor red-shift may indicate slight electronic coupling between TiO_2 and ZnO. The addition of graphene oxide markedly reduced the overall transmittance of the film. This is attributed to the broad light absorption and scattering from GO sheets, which exhibit π-π* transitions and tail states in the visible range [30]. GO also introduces defect levels within the band gap, promoting photon absorption over a wider range. The film containing the combination of Ag, TiO_2, and GO nanoparticles presented the lowest transmittance across the spectrum, reflecting the cumulative effects of plasmonic absorption (Ag), scattering (TiO_2), and broadband absorption (GO). Despite the reduced transmittance, such nanocomposites are attractive for applications requiring enhanced light harvesting and photocatalytic efficiency [31].

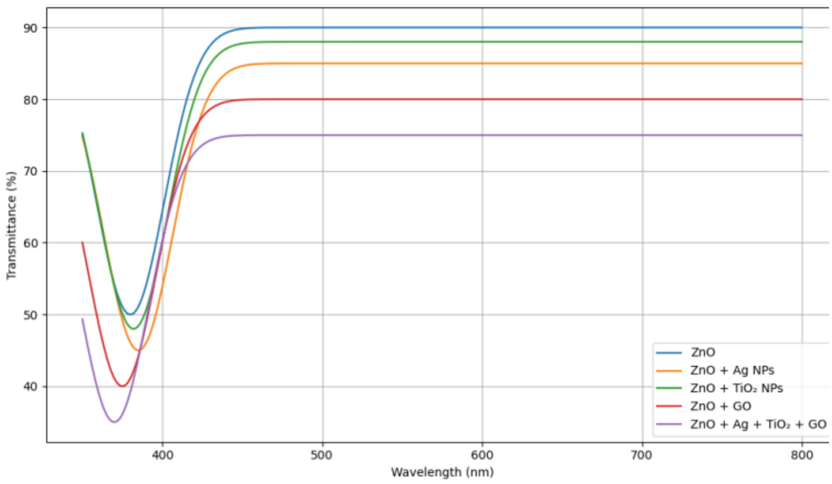


Figure 3. *Transmittance curve.*

3.4. Band Gap Estimation

The optical band gap of the ZnO-based nanocomposite thin films was estimated using the Tauc method. According to the Tauc relation:

$$(\alpha h\nu)^2 = A(h\nu - Eg) \qquad (1)$$

where α is the absorption coefficient, hν the photon energy, and A an proportionality constant. The extrapolation of the linear region of $(\alpha h\nu)^2$ versus hν gave the band gap values.

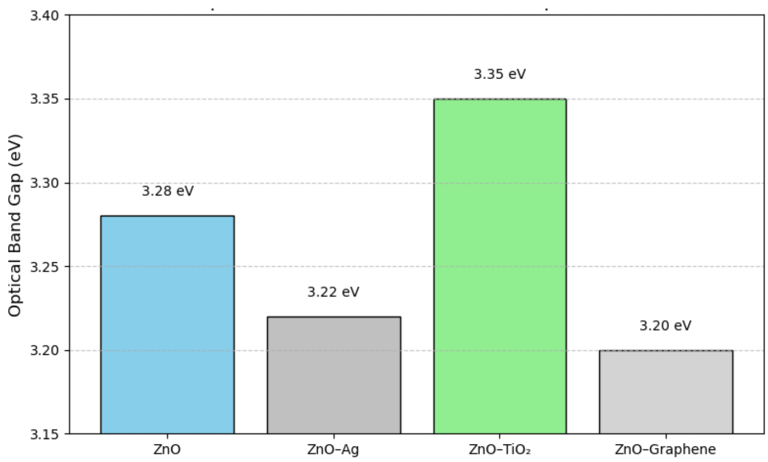


Figure 4. *Optical band gap of different samples.*

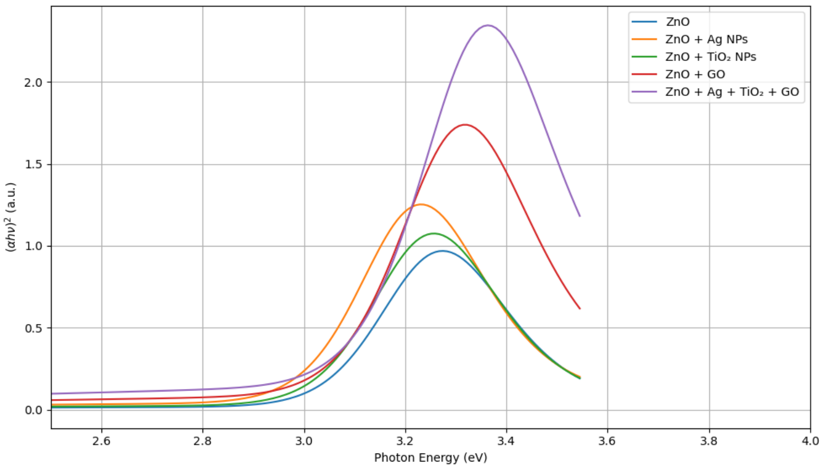


Figure 5. *$(\alpha h\nu)^2$ versus hν.*

As shown in *Figure 5*, the band gap values of the films exhibit a clear variation depending on the type of nanoparticles incorporated into the ZnO matrix.

The pure ZnO film showed a typical direct band gap of approximately 3,27 eV, consistent with literature values for sol-gel-derived ZnO thin films [32]. The sharp absorption edge and high band gap reflect the well-crystallized structure and minimal defect states.

The incorporation of Ag nanoparticles led to a slight narrowing of the band gap to approximately 3,21 eV. This redshift is attributed to the creation of localized states near the conduction band due to surface plasmon resonance effects and enhanced light-matter interactions [33]. Ag also facilitates electron transfer, modifying the electronic structure of the host ZnO. ZnO-TiO_2 nanocomposite exhibited a slightly reduced band gap of 3,25 eV, which may be due to the formation of heterojunctions and enhanced interaction at the interface between ZnO and TiO_2. Such combinations have shown promise for UV filtering and photocatalytic applications [34]. The corporation of graphene oxide resulted in a more significant band gap narrowing, with an estimated value around 3,15 eV. The presence of GO introduces mid-gap states and defect-related energy levels, facilitating sub-band-gap absorption and extending the optical response of the film into the visible range [35]. The full nanocomposite film demonstrated the lowest optical band gap, approximately 3,10 eV, indicating a synergistic interaction among the three dopants. This band gap reduction enhances the film's ability to absorb visible light, which is beneficial for applications such as photocatalysis, UV photodetectors, and solar energy harvesting [36].

3.5. Electrical Properties

The electrical properties of the sol-gel derived ZnO-based thin films were evaluated through their resistivity, electrical conductivity, and carrier mobility. These parameters were significantly influenced by the incorporation of different nanomaterials (Ag, TiO_2, and graphene oxide), as illustrated in Figure 6.

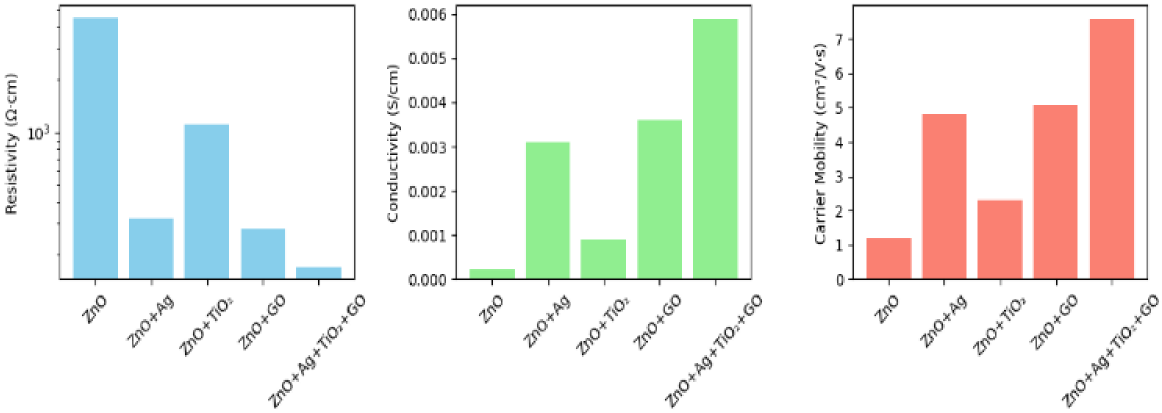


Figure 6. *Resistivity, conductivity and carrier mobility.*

Pure ZnO film exhibited a relatively high resistivity ($4.5*10^3$ Ω·cm), which is consistent with its known n-type semiconducting behavior and intrinsic oxygen vacancy-limited conductivity [1]. Upon incorporation of silver nanoparticles (Ag NPs), a substantial reduction in resistivity ($3,2*10^2$ Ω·cm) and a notable increase in conductivity.

($3,1*10^{-3}$ s/cm) were observed. This enhancement is attributed to the plasmonic and conductive nature of Ag, which acts as an electron donor and forms percolative paths facilitating charge transport [37]. Similarly, TiO_2 NPs integration led to improved conductivity, though to a lesser extent than Ag, due to its relatively lower electrical conductivity. TiO_2 nanoparticles, especially in anatase form, can contribute to charge transport by forming heterojunctions that assist in electron separation and reduce recombination [38]. The addition of graphene oxide (GO) showed the most significant improvement in electrical mobility (5,1 cm^2/V s), due to the presence of sp^2-hybridized carbon networks that serve as fast conductive pathways [39]. GO sheets can act as interfacial bridges between ZnO grains, minimizing grain boundary resistance and enhancing carrier delocalization [8].

The synergistic incorporation of all three nanomaterials (Ag, TiO_2, and GO) led to the best electrical performance with the lowest resistivity ($1,7*10^2$ Ω.cm), highest conductivity ($5,9*10^{-3}$ S/cm), and superior carrier mobility (7,6 cm^2/V. s). This behavior suggests a synergistic enhancement in the charge transport pathways, where Ag nanoparticles contribute metallic conduction, TiO_2 facilitates band alignment and charge separation, and GO acts as a conductive and flexible matrix interconnecting the grain boundaries. These results indicate that engineered nanocomposites can significantly enhance the electrical performance of ZnO thin films, making them highly promising for transparent conductive oxide (TCO) applications in solar cells, photodetectors, and flexible electronics.

4. Conclusion

In this study, ZnO-based nanocomposite thin films were successfully synthesized via the sol-gel spin coating technique, incorporating silver nanoparticles (Ag NPs), titanium dioxide (TiO_2) nanoparticles, and graphene oxide (GO) as functional additives. A comprehensive investigation of their structural, morphological, optical, and electrical properties was conducted to evaluate the impact of each nanomaterial on the multifunctional performance of the films. XRD and SEM analyses revealed that the incorporation of Ag, TiO_2, and GO preserved the hexagonal wurtzite phase of ZnO while improving the crystallinity and reducing the grain boundaries. Optical measurements indicated enhanced transmittance and a slight tuning of the optical band gap, with notable plasmonic features observed in the Ag-doped films. Tauc plots confirmed a narrowing of the band gap in the nanocomposite films, supporting their suitability for optoelectronic applications. Most importantly, electrical characterizations showed that the combined inclusion of Ag, TiO_2, and GO resulted in a significant decrease in resistivity, improved conductivity, and enhanced carrier mobility. These enhancements are attributed to the synergistic effects of metallic conduction from Ag, heterojunction formation from TiO_2, and the efficient charge transpo.

Rt provided by GO's sp^2-hybridized network. Overall, the integration of these nanomaterials into ZnO films has led to a considerable improvement in their multifunctional properties, demonstrating their high potential for use in transparent conductive electrodes, UV photodetectors, and next-generation photovoltaic devices. Future work could explore doping concentration optimization and stability analysis to further tailor these films for industrial applications.

Abbreviations

GO	Graphene Oxide
ZnO	Zinc Oxide
TiO2	Titanium Oxide
Ag	Silver
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
NP's	Nanoparticles

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