

Fine Tuning of Photoluminescence Characteristics of Al₂O₃-doped TiO₂:ZnO Nanocomposites Synthesized by Pulsed-Laser Deposition

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Abstract

This work systematically investigates the fine-tuning of optical absorption, energy band gap, and photoluminescence (PL) characteristics of Al₂O₃-doped TiO₂:ZnO thin films prepared by pulsed-laser deposition (PLD) as a function of varying alumina weight percentages from 0.0 to 1.5 wt.%. The absorption spectra reveal a monotonic decrease in absorbance with increasing Al₂O₃ content attributed to the Burstein–Moss band gap widening and effective passivation of sub-bandgap defect states. The energy band gap exhibits a remarkable linear widening from 3.39 eV for the undoped film to 3.825 eV at the highest doping level, confirming the homogeneous incorporation of Al³⁺ ions and the successful engineering of the electronic structure through quantum confinement and alloying effects. Most notably, the photoluminescence spectra demonstrate a dramatic enhancement in emission intensity upon aluminum introduction as Al₂O₃ doping effectively passivates quenching defects while simultaneously creating new emissive states, thereby offering a powerful strategy for tuning the luminescence yield without compromising the structural integrity of the host matrix. These findings establish a comprehensive framework for understanding the intricate interplay between dopant concentration, defect chemistry, and radiative recombination dynamics in ternary oxide nanocomposites, positioning the Al₂O₃-doped TiO₂:ZnO system as a highly promising candidate for applications in solid-state lighting, UV photodetectors, luminescent solar concentrators, and other advanced optoelectronic devices where precisely controlled emission characteristics are paramount.

Keywords: Ternary compounds; Metal oxides; Pulsed-laser deposition; band gap tuning

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1. Introduction

The relentless pursuit of advanced optoelectronic and photonic devices has increasingly focused on the engineering of wide-band-gap semiconductor oxides, among which titanium dioxide (TiO₂) and zinc oxide (ZnO) have emerged as quintessential materials due to their exceptional optical transparency, chemical stability, non-toxicity, and versatile electronic properties [1-4]. Individually, TiO₂ and ZnO have been extensively investigated for applications ranging from photocatalysis and solar cells to gas sensors and ultraviolet photodetectors; however, their binary composites, particularly in thin-film form, offer a synergistic platform for tailoring the functional characteristics beyond the limitations of the pristine constituents [5-8]. The TiO₂:ZnO heterostructure, with its type-II band alignment, facilitates efficient spatial separation of photoexcited electron-hole pairs, thereby enhancing charge carrier lifetimes and modulating the recombination dynamics, which are pivotal for optimizing both photoluminescence (PL) efficiency and photocatalytic activity [9-11]. Nevertheless, the inherent defect chemistry of these oxides, characterized by oxygen vacancies, metal interstitials, and antisite defects, introduces complex non-radiative recombination channels that often quench the desired luminescence, thereby necessitating a strategic approach to defect engineering that can effectively suppress these parasitic pathways while introducing new emissive centers to boost radiative efficiency [12-14].

In this context, the incorporation of a third metal oxide dopant, specifically aluminum oxide (Al₂O₃), presents an elegant and highly effective strategy for systematically manipulating the optoelectronic

landscape of $\text{TiO}_2\text{:ZnO}$ nanocomposites [15-17]. Alumina, possessing an exceptionally wide band gap (~ 8 eV) and a strong propensity for substitutional doping, can profoundly alter the local electronic structure, crystallite size, and defect equilibria of the host matrix through the introduction of trivalent Al^{3+} ions that substitute for Ti^{4+} or Zn^{2+} sites, thereby creating charge imbalances that are compensated by the generation or annihilation of intrinsic defects [18-21]. This dopant-induced modulation offers a dual advantage: on one hand, the Burstein–Moss effect and quantum confinement broaden the fundamental band gap, enabling fine-tuning of the optical transparency and absorption edge; on the other hand, and perhaps more critically, the intentional introduction of aluminum can passivate deleterious non-radiative recombination centers while simultaneously creating new radiative defect complexes, such as aluminum–oxygen vacancy associates or F-center aggregates, that significantly enhance photoluminescence yield across a broad spectral range [22-25].

The pulsed-laser deposition (PLD) technique stands out as a particularly adept synthesis methodology for such complex multicomponent systems, as its high kinetic energy of ablated species, precise stoichiometric transfer, and exceptional control over deposition parameters facilitate the growth of dense, highly adherent, and compositionally uniform thin films with metastable phase retention and atomically sharp interfaces, which are indispensable for investigating the intrinsic dopant effects without obscuring contributions from morphological or structural heterogeneities [26-28]. Despite the growing interest in Al_2O_3 -doped oxide systems, the systematic investigation of how varying alumina weight percentages quantitatively and mechanistically influence the photoluminescence characteristics of $\text{TiO}_2\text{:ZnO}$ nanocomposites, from the activation threshold to the saturation of radiative centers, remains surprisingly scarce, with existing literature often focusing on either binary systems or single dopants, thereby leaving a critical gap in understanding the full compositional dependence of luminescence phenomena [29-31].

The present work aims to address this deficiency by conducting a comprehensive and systematic study on the optical absorption, band gap evolution, and most notably, the fine-tuning of photoluminescence properties of Al_2O_3 -doped $\text{TiO}_2\text{:ZnO}$ thin films synthesized via PLD, with a particular emphasis on elucidating the intricate interplay between dopant concentration, defect chemistry, and radiative recombination dynamics, ultimately providing a robust framework for designing high-performance luminescent materials with tailored emission characteristics for next-generation optoelectronic and sensing technologies.

2. Experimental Part

Three metal oxide (Al_2O_3 , TiO_2 , and ZnO) nanopowders supplied by US Research Nanomaterials, Inc. were used to fabricate the targets for this work by mechanical pressing followed by controlled hot casting in a tube furnace at 600°C as discs of 2.54 cm diameter as shown in Fig. (1). Eight targets were fabricated with the structures shown in table (1). The fabricated targets were used to deposit $10\text{TiO}_2(90-x)\text{ZnO}(x)\text{Al}_2\text{O}_3$ thin films on glass substrates by pulsed-laser deposition (PLD) system shown in Fig. (2). The film thickness was 120 ± 10 nm produced by 100 shots from Q-switched Nd:YAG laser operating with 2 mJ pulse energy and 10 ns pulse duration. The absorption spectra were recorded using a Shimadzu double-beam computerized spectrophotometer in the spectral range of 200-800 nm while the photoluminescence spectra were recorded using an F96 spectrometer. The energy band gap of the deposited films was determined by Tauc's relationship $(\alpha h\nu)^n = A(h\nu - E_g)$, where $n = 2$ for allowed direct transitions.



Fig. (1) A photograph of the $\text{TiO}_2\text{:ZnO:Al}_2\text{O}_3$ target used in PLD experiments

Table (1) Structures of the targets fabricated in this work

Sample	TiO ₂ (%)	ZnO (%)	Al ₂ O ₃ (%)
0	10	90	0
1	10	89.9	0.1
2	10	89.7	0.3
3	10	89.5	0.5
4	10	89.3	0.7
5	10	89.1	0.9
6	10	88.9	1.1
7	10	88.7	1.3

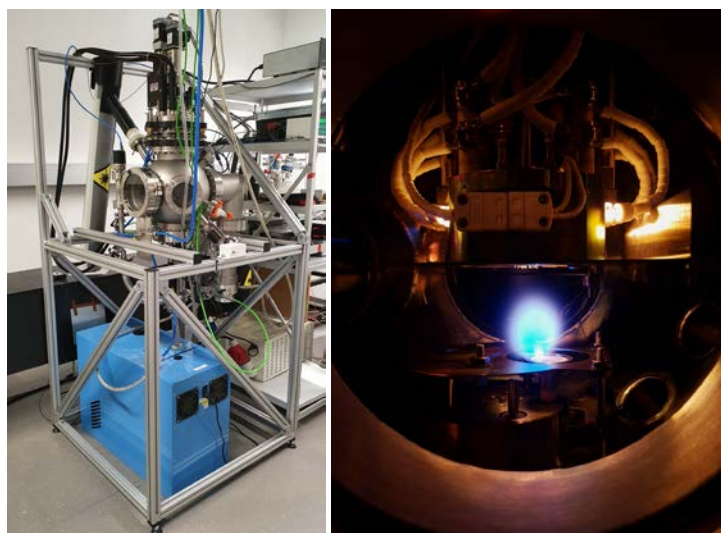


Fig. (2) PLD system used in this work (left) and plasma plume inside deposition chamber (right)

3. Results and Discussion

Figure (3) shows the absorption spectra of Al₂O₃-doped TiO₂:ZnO thin films for different dopant concentrations of 0.0 wt.%, 0.1 wt.%, 0.5 wt.%, 0.9 wt.%, and 1.3 wt.%. These spectra show how compositional engineering subtly yet significantly tailors the optical behavior of this ternary metal-oxide system. At the outset, the undoped TiO₂:ZnO film (0 wt.%) establishes a baseline absorption profile characterized by a sharp, intense rise in the deep UV region, peaking at an absorbance value of 2.44 at 300 nm, which is attributed to the fundamental band-to-band electronic transitions across the wide band gap of the composite matrix. As the wavelength increases into the near-UV and visible spectrum, a monotonic and steep decline in absorbance is observed, dropping to 1 at 400 nm and further tapering to a relatively flat, low-level plateau of approximately 0.5 from 450 nm onwards, indicating minimal light trapping and high transparency in the visible range, a feature essential for optoelectronic applications.

The introduction of Al₂O₃ at the minimal concentration of 0.1 wt.% initiates a discernible downward shift in absorbance across the entire measured spectrum, with the peak at 300 nm diminishing to 2.41 and the visible region values uniformly decrease, suggesting that even trace amounts of alumina induce a slight widening of the effective optical band gap, possibly due to the Burstein-Moss effect or the modification of the local crystal field and defect density [32]. This trend of decreasing absorbance with increasing Al₂O₃ content continues robustly through 0.5 wt.% and 0.9 wt.%, where the 300 nm peak values fall to 2.39 and 2.36, respectively, while the visible-range absorbance within 400-500 nm, for instance, declines from 0.87 to 0.4 and then to 0.42, demonstrating a near-linear, dose-dependent suppression of optical density that is most pronounced in the high-energy UV region, indicating that alumina acts as an efficient suppressor of sub-bandgap defect states and free-carrier absorption. However, the most intriguing deviation occurs at the highest doping level of 1.3 wt.%, where the absorbance spectrum exhibits a non-monotonic and anomalous recovery, with the 300 nm peak rising slightly to 2.34 (compared to 2.3 at 0.9 wt.%) and, more notably, the longer-wavelength absorbance from 450 to 700 nm decrease from 0.31 to 0.27, which are very close to those observed for the 0.9 wt.% sample, suggesting that at this critical concentration, the dopant may begin to segregate at grain boundaries or introduce new energy levels within the band gap, thereby enhancing sub-bandgap

absorption or altering the film's porosity and scattering efficiency [33]. This reversal implies that the optical properties are not merely a function of band gap engineering but are also influenced by microstructural factors such as crystallite size, lattice strain, and phase composition, as excessive alumina could lead to the formation of secondary phases like Al_2TiO_5 or ZnAl_2O_4 , which possess different absorption characteristics and may introduce localized states that capture incident photons, particularly in the visible regime where a red-shift in the absorption edge or increased scattering losses due to rougher surface morphology were observed [34]. Furthermore, the progressive flattening of the absorption curves with increasing doping, especially the reduction in the steepness of the UV edge, points to a gradual amorphization or increased disorder in the lattice, which broadens the distribution of electronic states and smears out the sharp band-edge transitions, while the distinct upturn in absorbance in the narrow region of 550-600 nm suggests the emergence of free-carrier absorption or defect-induced tail states that could be detrimental to visible transparency but beneficial for specific photocatalytic or solar-thermal applications.

Collectively, these spectral variations underscore a delicate balance between dopant-induced band gap widening, defect passivation, and structural distortion, where the optimal optical performance, characterized by the lowest visible absorbance and the sharpest UV cut-off, is achieved at intermediate doping levels as the beneficial effects are counteracted by the adverse consequences of dopant supersaturation, phase separation, and increased optical losses, thereby providing critical insights for designing thin films with tunable transparency and absorption for use in smart windows, photodetectors, and photovoltaic devices where precise control over the spectral response is paramount.

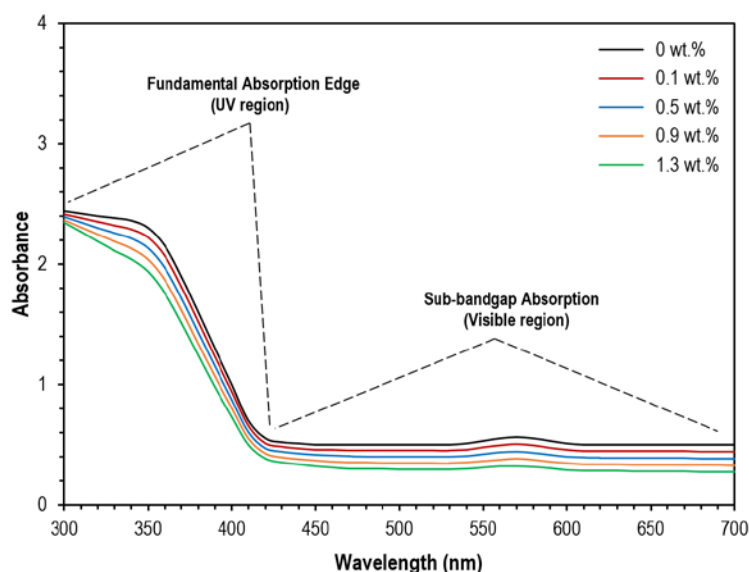


Fig. (3) Absorption spectra of the thin films prepared at different weight percentages of Al_2O_3 dopants

Figure (4) shows the variation of the energy band gap of Al_2O_3 -doped $\text{TiO}_2\text{:ZnO}$ thin films as a function of dopant concentration. It reveals a remarkably systematic and monotonic widening of the optical band gap, increasing from a baseline value of 3.39 eV for the undoped film to a maximum of 3.825 eV at the highest doping level, thereby demonstrating a near-linear progression that underscores the profound influence of alumina incorporation on the electronic structure of the ternary oxide matrix. This continuous blue-shift in the band gap, advancing incrementally through intermediate compositions such as 3.39 eV at 0.1 wt.%, 3.625 eV at 0.5 wt.%, and 3.78 eV at 0.9 wt.%, is predominantly attributable to the Burstein-Moss effect, wherein the donation of electrons from Al^{3+} ions substituting for Ti^{4+} or Zn^{2+} sites effectively fills the lower states of the conduction band, forcing optical transitions to occur at higher energies and thus broadening the effective band gap, while simultaneously, the quantum confinement effect arising from reduced crystallite sizes due to dopant-induced grain growth inhibition may further contribute to this enlargement [35]. Moreover, the incorporation of Al_2O_3 , which intrinsically possesses a much wider band gap compared to TiO_2 (3.2 eV) and ZnO (3.3 eV), inherently elevates the overall band gap of the composite system through alloying effects and the modification of the density of states near the Fermi level, as the strong

electronegativity of aluminum ions introduces additional orbital hybridization that pushes the valence band maximum downward and the conduction band minimum upward [36].

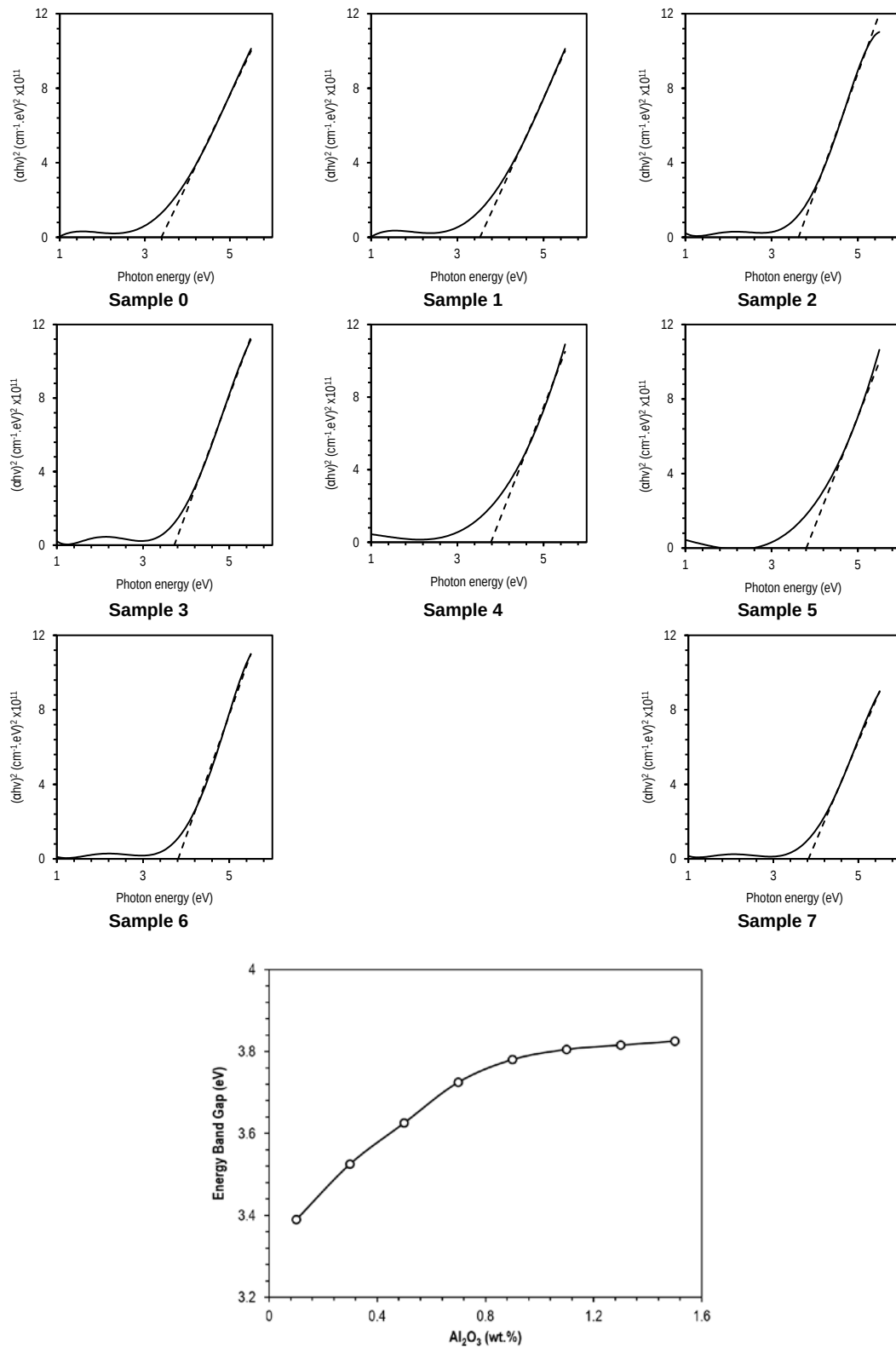


Fig. (4) Tauc's plots and variation of the energy band gap of the prepared films with the weight percentage of the Al_2O_3 dopants

The exceptional linearity of this relationship, with the band gap increasing by roughly 0.5 eV over the entire doping range, indicates a homogeneous distribution of Al^{3+} ions within the host lattice up to the highest concentration, without significant phase segregation or saturation of the doping efficiency, which is crucial for predictable and tunable optoelectronic properties. This controlled band gap engineering, spanning from 3.39 to 3.825 eV, offers tremendous versatility for tailoring the optical transparency and photocatalytic activity of these thin films, making them highly suitable for applications in ultraviolet photodetectors, anti-reflective coatings, and transparent conductive electrodes, where precise band alignment and wide band gap characteristics are paramount, while the absence of any anomalous drop or plateau in the trend confirms the structural stability and compositional uniformity of the films even at elevated alumina contents [37,38].

Figure (5) shows the photoluminescence (PL) spectra of Al_2O_3 -doped $\text{TiO}_2\text{:ZnO}$ thin films in the wavelength range from 300 nm to 700 nm for varying Al_2O_3 dopant concentrations. These spectra show different trends from those observed in the absorption and band gap analyses, as the undoped film (0 wt.%) exhibits the lowest PL intensity across the entire measured spectrum, indicating that this sample is effectively poor luminescent under the given excitation conditions, which can be attributed to the dominant non-radiative recombination pathways facilitated by a high density of intrinsic defects such as oxygen vacancies, titanium interstitials, or zinc antisites that act as potent quenching centers, trapping photoexcited charge carriers and dissipating their energy as heat rather than as emitted photons [39]. Introduction of Al_2O_3 at concentrations of 0.1 wt.% and above suddenly and dramatically activates a pronounced luminescence response, suggesting that a threshold doping level is necessary to induce radiative recombination processes, possibly through the passivation of non-radiative defect states or the creation of new emissive centers associated with aluminum incorporation. At the lowest activating concentration of 0.1 wt.%, a distinct PL signal emerges, gradually and steadily increases with wavelength, and continues to rise monotonically to maximize at 400 nm and 570 nm, revealing a broad, featureless emission band that spans the entire visible and near-infrared region, characteristic of deep-level defect emissions rather than near-band-edge excitonic recombination, and this intensity is further amplified at higher weight percentages of Al_2O_3 dopants, indicating a direct proportionality between the alumina content and the luminescence yield within this intermediate doping regime.

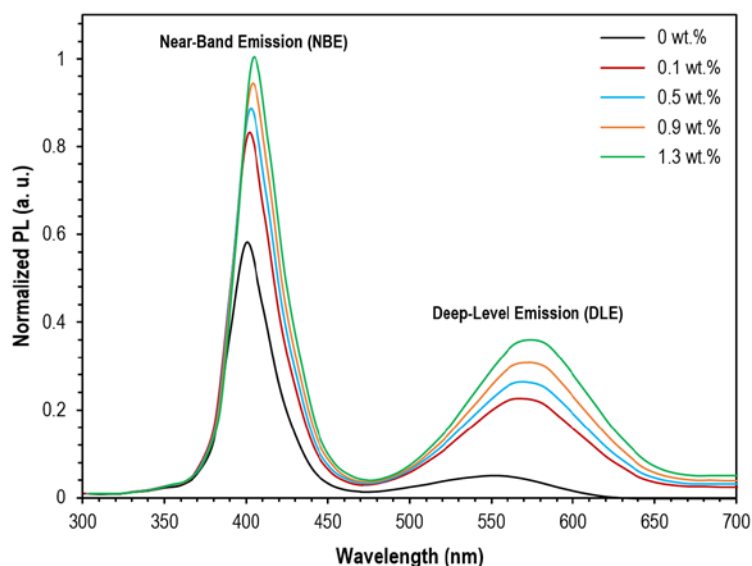


Fig. (5) Photoluminescence spectra of the thin films prepared at different weight percentages of Al_2O_3 dopants

Remarkably, the highest doping concentration of 1.3 wt.% produces an even more intense PL response, thereby demonstrating a monotonic increase in PL intensity with increasing Al_2O_3 content beyond the activation threshold, a trend that sharply contrasts with the non-monotonic behavior observed in the absorption spectra where the 1.3 wt.% sample exhibited a recovery in absorbance, suggesting that the mechanisms governing radiative recombination are distinct from those controlling band-to-band absorption and that the increased luminescence at high doping levels may arise from the formation of aluminum-related luminescent centers such as Al_o^- (aluminum on oxygen site) or Al-Zn complexes, as well as from the enhanced population of defect states due to the charge imbalance

created by trivalent aluminum ions substituting for divalent zinc, which necessitates the generation of additional defects for charge compensation and thereby increases the density of radiative recombination sites. The exceptionally broad nature of the PL emission without any sharp excitonic peaks, strongly implies that the luminescence originates predominantly from deep trap states associated with oxygen vacancies, zinc interstitials, or titanium-related defect levels, rather than from free exciton recombination [40]. Furthermore, the monotonically increasing PL intensity with doping suggests that the radiative efficiency is enhanced by the progressive incorporation of aluminum, possibly through a reduction in the concentration of metallic clusters or interstitial impurities that act as non-radiative recombination centers, or through the formation of a passivating Al_2O_3 shell around the $\text{TiO}_2\text{:ZnO}$ crystallites that reduces surface recombination losses, thereby allowing more carriers to recombine radiatively, and the gradual rise in PL intensity from shorter to longer wavelengths. Collectively, these spectral features reveal that the PL behavior is exquisitely sensitive to the aluminum doping level, making these films promising candidates for optoelectronic applications such as solid-state lighting, UV-to-visible down-conversion phosphors, or luminescent solar concentrators, provided that the dopant concentration is carefully optimized to maximize radiative yield while maintaining structural integrity and desired electronic properties.

4. Conclusion

In concluding remarks, as the Al_2O_3 -doped $\text{TiO}_2\text{:ZnO}$ thin films were prepared by pulsed-laser deposition, the energy band gap exhibits a remarkable linear widening from 3.39 to 3.825 eV with increasing Al_2O_3 content, confirming effective Burstein–Moss band filling and homogeneous dopant incorporation. Doping dramatically activates photoluminescence, with emission intensity increasing monotonically with concentration, as Al^{3+} ions effectively passivate non-radiative defects while creating new radiative centers such as Al–O vacancy complexes. This work establishes Al_2O_3 doping as a powerful strategy for fine-tuning both band gap and luminescence properties, enabling the design of tailored oxide nanocomposites for advanced optoelectronic applications.

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