

RESEARCH ARTICLE

Annealing Improvement in Structural, Optical and Photoluminescence Properties of Eu - Doped ZnO Films

Anil U. Sonawane^{1*}, Chetan K. Kasar², Tahira Mir³

ABSTRACT: In this study, 10 at.% Europium (Eu)-doped ZnO nanofilms were deposited on microscopic glass substrates using the spin-coating technique and subsequently annealed at temperatures ranging from 250 to 400°C. The effect of post-annealing on the structural, optical, and photoluminescence properties of the films was investigated. Structural parameters, including crystallite size and lattice strain, were analysed using X-ray diffraction (XRD), which confirmed the formation of a hexagonal wurtzite ZnO structure. Optical studies performed using UV-Vis spectroscopy revealed high transparency in the visible region. An increase in optical absorption and a reduction in the optical band gap were observed with increasing annealing temperature. Photoluminescence (PL) spectra exhibited a dominant ultraviolet (UV) emission peak along with weak visible emission bands. The UV emission peak showed a red shift (shift toward longer wavelengths) with increasing annealing temperature, indicating improved crystallinity and a reduction in defect-related states. These findings demonstrate that post-annealing plays a crucial role in tailoring the optoelectronic properties of Eu-doped ZnO nanofilms, making them promising candidates for luminescent and optoelectronic device applications.

Keywords: Eu-Doped ZnO, Thin Films, Post-Annealing, Optical Band Gap, Photoluminescence

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1. INTRODUCTION

Direct-band-gap semiconductor nanomaterials have attracted significant research interest over recent decades because of their remarkable nanoscale properties. Compared with their bulk counterparts, semiconductor nanocrystals often exhibit tunable band gaps and enhanced luminescence efficiency due to quantum confinement effects [1, 2]. Among the II–VI semiconductor materials, zinc oxide (ZnO)

has received considerable attention owing to its excellent optoelectronic properties. ZnO is a wide-direct-band-gap semiconductor with a band gap energy of 3.37 eV and a large exciton binding energy of 60 meV at room temperature [3, 4]. These properties make ZnO a promising material for various optoelectronic and photonic applications, including blue light-emitting diodes, short-wavelength laser diodes, transparent conducting electrodes, surface acoustic wave devices, and chemical and biological sensors [5].

One of the most effective approaches to improve the properties of ZnO nanomaterials is doping with rare-earth (RE) elements [6]. Rare-earth ions possess partially filled 4f electronic shells that are shielded by the outer 5s and 5p orbitals, resulting in optical transitions that are relatively insensitive to the surrounding crystal field. When incorporated into direct-band-gap semiconductor hosts, RE ions act as luminescent centers, producing intense emission

¹ Department of Electronics, DNCVPS Shirish Madhukarrao Chaudhari College, Jalgaon, 425001 Maharashtra, India.

² St. Joseph's Convent Junior College, Jalgaon, 425001 Maharashtra, India.

³ Department of Electronics, Smt. G. G. Khadse College, Muktainagar, Jalgaon, Maharashtra, 425001 India.

* Author to whom correspondence should be addressed:
ausonawane88@gmail.com (A. U. Sonawane)

and narrow spectral lines [7, 8]. Among the various rare-earth dopants, Lanthanum (La), Cerium (Ce), Ytterbium (Yb), and Europium (Eu) have been widely investigated. Eu-doped ZnO has emerged as a particularly attractive material because of its unique structural and optical characteristics [9]. It has potential applications in optoelectronic devices such as light-emitting diodes, solar cells, display devices, and sensors [10]. Europium can exist in two oxidation states, Eu^{2+} and Eu^{3+} , each exhibiting distinct luminescence behavior. Eu^{2+} ions typically produce broad blue-to-green emission bands, whereas Eu^{3+} ions generate sharp red emission lines arising from intra-4f electronic transitions. The difference in ionic radii between Zn^{2+} (74 pm) and Eu^{3+} (90 pm) can introduce lattice distortions and defect states in the ZnO crystal structure, significantly influencing its luminescence characteristics [11, 12]. Furthermore, the relative position of Eu^{3+} charge-transfer levels with respect to the ZnO conduction band and the mismatch between exciton lifetime and Eu^{3+} decay time can affect the efficiency of energy transfer processes [13]. The Eu concentration was fixed at 10 at.% in the present study based on previous reports indicating that this concentration provides enhanced luminescence intensity while maintaining the crystalline quality of ZnO. At lower Eu concentrations, the number of luminescent centers may be insufficient to produce strong emission, whereas excessive Eu incorporation can lead to concentration quenching, defect formation, secondary phase segregation, and deterioration of crystal quality. Therefore, a concentration of 10 at.% was selected to investigate the influence of post-annealing temperature on the structural and optical properties of Eu-doped ZnO nanofilms [14–16].

Several studies have reported that the luminescence efficiency of Eu-doped ZnO nanostructures can be enhanced by optimizing synthesis conditions, particle size, and post-treatment processes such as thermal annealing [8]. Post-deposition annealing plays a crucial role in modifying defect structures, improving crystallinity, promoting grain growth, and facilitating dopant incorporation into the ZnO lattice. Therefore, controlling annealing temperature and processing conditions is essential for tailoring the structural and optical properties of Eu-doped ZnO nanomaterials [17, 18]. Various techniques have been employed for the deposition of rare-earth-doped ZnO thin films, including spray pyrolysis, magnetron sputtering, hydrothermal synthesis, thermal evaporation, chemical vapor deposition, and sol-gel processing [19]. Among these methods, the sol-gel spin-coating technique offers several advantages, including low processing temperature, cost-effectiveness, excellent

compositional control, and the ability to produce uniform thin films over large areas [20]. In the present work, 10 at.% Eu-doped ZnO nanofilms were deposited on glass substrates using the sol-gel spin-coating technique and subsequently annealed at temperatures ranging from 250 to 400 °C. The influence of post-annealing temperature on the structural, optical, and photoluminescence properties of the films was systematically investigated. The crystalline structure was analyzed using X-ray diffraction (XRD), while optical properties were examined using UV-Visible spectroscopy and photoluminescence measurements. The effects of annealing temperature on crystallinity, optical transparency, band-gap energy, and luminescence characteristics were analyzed in detail. The results demonstrate that post-annealing significantly influences the optoelectronic properties of Eu-doped ZnO nanofilms, highlighting their potential for advanced photonic and optoelectronic device applications.

2. EXPERIMENTAL DETAILS

Europium-doped zinc oxide (Eu) nanofilms containing 10 at.% Eu were fabricated on microscope glass substrates (2.5 cm × 1.5 cm) via the sol-gel spin-coating technique. Zinc acetate dihydrate [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, (Sigma-Aldrich, 99.99% purity)] and europium nitrate pentahydrate [$\text{Eu}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, (Sigma-Aldrich, 99.99% purity)] were employed as Zn and Eu precursor sources, respectively. 2-Methoxyethanol ($\text{C}_3\text{H}_8\text{O}_2$) served as the solvent, while ethanolamine ($\text{H}_2\text{NCH}_2\text{CH}_2\text{OH}$) was used as a stabilizing and complexing agent.

A 0.5 M precursor solution was prepared by dissolving the appropriate amount of zinc acetate dihydrate in 30 mL of 2-methoxyethanol under continuous magnetic stirring at 70 °C for 30 min. Subsequently, europium nitrate pentahydrate was introduced into the solution to achieve a dopant concentration of 10 at.%. Thereafter, 5 mL of ethanolamine was added dropwise, and the resulting solution was further stirred at 75 °C for 25 min until a clear and homogeneous sol was obtained. The prepared sol was then aged at room temperature for 10 h to ensure complete hydrolysis and stabilization of the precursor species.

Prior to deposition, the microscope glass substrates were thoroughly cleaned using an ultrasonic bath with ethanol, distilled water, and acetone in sequence to eliminate surface contaminants and improve film adhesion. The aged precursor solution was deposited onto the cleaned substrates using a spin-coating system operated at a rotational speed of 2000 rpm

for 30 s. To obtain the desired film thickness, the coating process was repeated twelve times. Following each deposition cycle, the films were subjected to intermediate heat treatment at 160 °C for 8 min to evaporate residual solvents and initiate the decomposition of organic constituents.

After completion of the multilayer deposition process, the films were annealed in ambient air for 1 h at different temperatures ranging from 250 to 400 °C. A reference sample was retained at 160 °C without further thermal treatment. The post-deposition annealing process promotes crystallization of the ZnO phase, facilitates the incorporation of Eu ions into the ZnO lattice, reduces residual organic impurities, and modifies the concentration of intrinsic defects. Consequently, the influence of annealing temperature on the structural, optical, and photoluminescence characteristics of the Eu-doped ZnO nanofilms was systematically investigated.

3. RESULTS AND DISCUSSION

3.1 Structural Properties

The structural properties of the 10 at.% Eu-doped ZnO thin films prepared at 160 °C and subsequently annealed at temperatures ranging from 250 to 400 °C were investigated using X-ray diffraction (XRD). The corresponding diffraction patterns are presented in Figure 1.

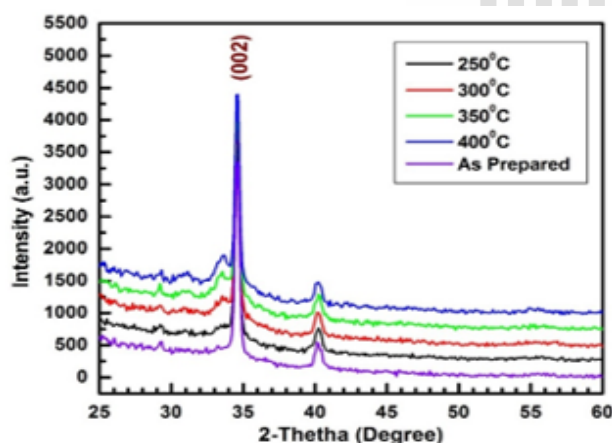


Fig. 1. XRD spectra of 10 at.% Eu doped ZnO films prepared, and post annealed at different temperatures.

All films exhibit similar diffraction profiles without any significant shift in the diffraction peak positions (2θ), indicating that the crystal structure remains stable after thermal annealing. The diffraction peaks are in good agreement with the standard hexagonal

wurtzite ZnO structure (JCPDS Card No. 36-1451) [21], confirming the formation of a single-phase ZnO crystalline structure. No secondary phases related to europium oxides or other impurity phases were detected within the detection limit of the XRD technique. This observation suggests that Eu ions are either incorporated into the ZnO lattice or are present below the XRD detection limit without significantly altering the host crystal structure.

A prominent diffraction peak centered at $2\theta \approx 34.40^\circ$ corresponding to the (002) crystallographic plane was observed for all samples. The strong intensity of the (002) reflection indicates a preferred orientation along the c-axis perpendicular to the substrate surface. Such preferential growth is characteristic of high-quality ZnO thin films and is advantageous for optoelectronic applications. An increase in the intensity of the (002) peak with increasing annealing temperature was observed, particularly for the film annealed at 400 °C. Simultaneously, the diffraction peaks became sharper and more intense, indicating an improvement in crystallinity. The enhancement in crystallinity can be attributed to the reduction of structural defects, release of internal stresses, and rearrangement of atoms into energetically favorable lattice positions during thermal treatment. In contrast, the film annealed at lower temperature exhibited broader diffraction peaks, indicating a higher defect density and lower crystalline quality [22]. The average crystallite size (D) and microstrain (ε) were estimated from the full width at half maximum (FWHM) of the dominant (002) diffraction peak using Scherrer's equation (1) and the lattice strain relation (2): [23].

$$D = \frac{0.94 \lambda}{\beta \cos \theta} \quad (1)$$

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (2)$$

Where λ is the wavelength of the incident X-ray radiation, β is the full width at half maximum (FWHM) expressed in radians, and θ is the Bragg diffraction angle.

The calculated crystallite sizes for the films annealed at 250, 300, 350, and 400 °C were found to be 40.59, 39.63, 38.71, and 37.83 nm, respectively. The corresponding micro-strain values increased from 0.853923×10^{-3} to 0.937232×10^{-3} with increasing annealing temperature, as summarized in Table 1.

The slight decrease in crystallite size accompanied by an increase in micro-strain may be attributed to lattice distortion induced by the incorporation of Eu

Table 1. Structural and optical parameters of Eu-doped ZnO nanofilms deposited on glass substrate.

Post Annealing Temperature (°C)	Crystallite Size (nm)	FWHM	Micro Strain (10^{-3})	PL Wavelength (nm)	Optical Band Gap (eV)
As Prepared	40.59	0.205	0.853923	380	3.360
250	39.63	0.210	0.874750	387	3.310
300	38.71	0.215	0.895577	390	3.280
350	36.98	0.225	0.916405	394	3.275
400	37.83	0.220	0.937232	395	3.260

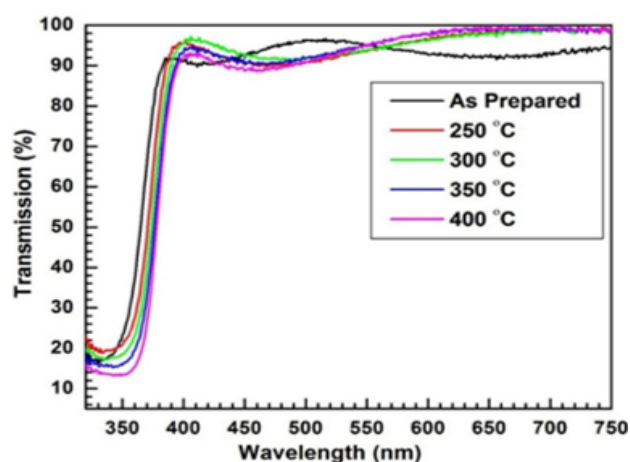
ions and thermally activated defect redistribution during annealing.

Although the crystallite size exhibits a marginal reduction, the increased diffraction peak intensity indicates an overall improvement in crystalline ordering and preferential orientation. Therefore, the observed enhancement in XRD peak intensity suggests improved structural quality of the films following thermal treatment. The structural evolution observed from XRD analysis correlates well with the optical and photoluminescence results, demonstrating that post-annealing plays a significant role in tailoring the crystallinity and defect structure of Eu-doped ZnO thin films.

3.2 Optical Properties

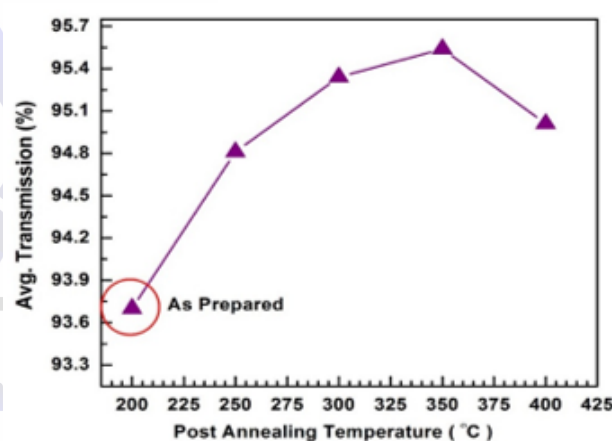
3.2.1 Optical Properties: UV-Vis Spectra

The optical transmittance spectra of 10 at.% Eu-doped ZnO nanofilms deposited on glass substrates and annealed at different temperatures in ambient air are presented in Figure 2. All films exhibit a sharp transmission edges and high optical transparency exceeding 90% in the visible region, indicating good optical quality and low optical losses [24]. The annealed films exhibit higher transmittance than the as-prepared film, which may be attributed

**Fig. 2.** Transmission Spectra of Post annealed nanofilms.

to improved crystallinity and a reduction in defect- and impurity-related scattering centers. Thermal annealing promotes the decomposition of residual organic species and enhances structural ordering, thereby improving optical transmission through the films.

Figure 3 shows the variation of average transmittance as a function of annealing temperature. The average transmittance increases gradually with increasing annealing temperature, indicating an improvement in film quality.

**Fig. 3.** Average transmission of Eu doped ZnO nanofilms at different Post annealed temperature.

However, a slight reduction in transmittance is observed for the film annealed at 400 °C compared with that annealed at 350 °C. This behavior may be associated with increased optical scattering, defect redistribution, or microstructural modifications induced by high-temperature annealing.

3.2.2 Absorption spectra

Figure 4 shows the UV-Visible absorption spectra of the as-prepared and annealed Eu-doped ZnO nanofilms recorded in the wavelength range of 300–750 nm. All samples exhibit strong absorption in the ultraviolet region (approximately 320–375 nm), which is characteristic of wide-band-gap ZnO semiconductors. The presence of a sharp absorption

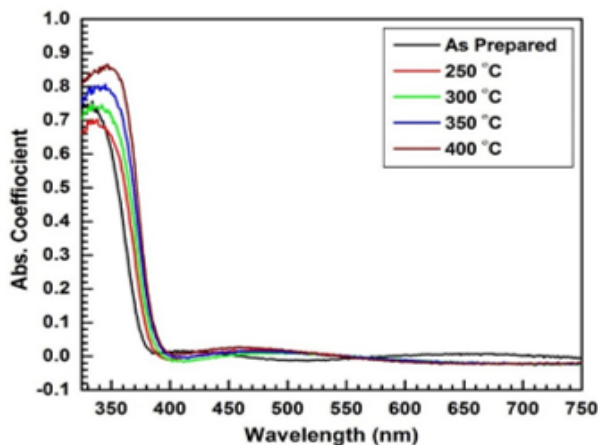


Fig. 4. Absorbance spectra of Eu doped ZnO nanofilms at different Post annealed temperature.

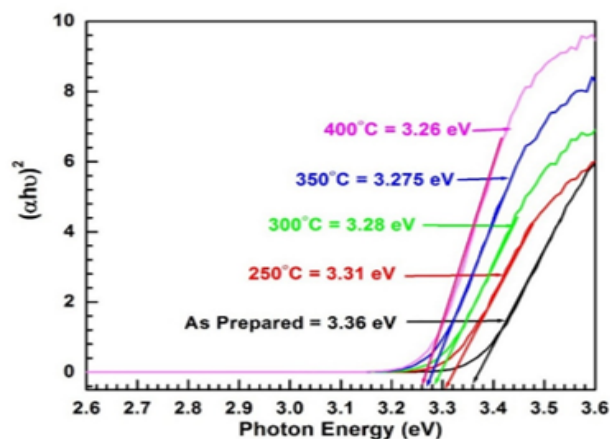


Fig. 5. Absorption coefficient vs photon energy of Eu doped ZnO films annealed at different temperatures.

edge confirms the formation of high-quality ZnO-based thin films with a well-defined electronic band structure [25]. An increase in absorption intensity is observed with increasing annealing temperature. The enhancement in optical absorption may be attributed to improved crystallinity, modification of defect states, and increased interaction between incident photons and the film material. The film annealed at 400 °C exhibits the highest absorption intensity among all samples, indicating significant changes in its optical characteristics following thermal treatment [26]. In the visible region (400–750 nm), all films exhibit very low absorption and correspondingly high transparency, which are desirable characteristics for transparent optoelectronic applications. The combination of strong ultraviolet absorption and high visible transparency suggests that Eu-doped ZnO nanofilms are promising materials for ultraviolet photodetectors, transparent electronic devices, light-emitting diodes, and other advanced optoelectronic applications [12].

3.2.3 Optical Band Gap

The effect of post-annealing temperature on the optical band gap of the 10 at.% Eu-doped ZnO nanofilms is illustrated in Figure 5. The optical band gap energy (E_g) was determined using the Tauc relation for direct band-gap semiconductors [27]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (3)$$

Where α is the absorption coefficient, $h\nu$ is the photon energy, A is a proportionality constant, and E_g is the optical band-gap energy.

The optical band-gap energy was estimated by extrapolating the linear portion of the Tauc plots to the photon-energy axis. The calculated band-gap values decreased from 3.360 eV to 3.260 eV with increasing annealing temperature. The gradual reduction in band-gap energy indicates a red shift of the absorption edge with increasing annealing temperature. This behavior may be attributed to structural modifications induced by thermal treatment, including improved crystallinity, lattice distortion caused by Eu incorporation, defect-state evolution, and changes in localized energy levels within the ZnO band structure. The decrease in band-gap energy may also be associated with increased defect-related states near the band edges, which facilitate optical transitions at lower photon energies [28, 29].

3.3 Photoluminescence Properties

Figure 6 shows the room - temperature photoluminescence (PL) spectra of 10 at.% Eu-doped ZnO thin films annealed at different temperatures. T

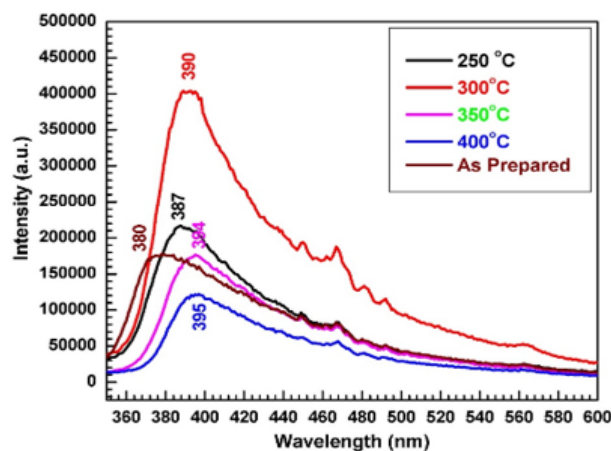


Fig. 6. PL spectra of Eu doped ZnO films annealed at different temperatures.

he PL measurements were performed using an excitation wavelength of 330 nm. All samples exhibit a strong ultraviolet (UV) emission band in the wavelength range of 380–395 nm, which is attributed to the near-band-edge (NBE) excitonic recombination of ZnO. In addition, a relatively weak broad visible emission band is observed in the range of 450–495 nm, which is commonly associated with intrinsic defects in ZnO, such as oxygen vacancies (V_o), zinc interstitials (Zn_i), and other defect-related states [30].

The as-prepared and low-temperature annealed films exhibit relatively weak PL intensity, indicating the presence of a higher density of structural defects and non-radiative recombination centers. Upon annealing at 300 °C, a significant enhancement in the NBE emission intensity is observed, with the dominant emission peak centered near 390 nm. The increase in PL intensity can be attributed to improved crystallinity, reduced defect concentration, and suppression of non-radiative recombination pathways resulting from thermal annealing. These observations are consistent with the XRD results, which indicate improved structural ordering after annealing [31].

A gradual red shift of the NBE emission peak from approximately 380 nm to 395 nm is observed with increasing annealing temperature. This shift may be associated with band-gap narrowing, relaxation of internal strain, defect-state evolution, and modifications of the local electronic structure induced by Eu incorporation and thermal treatment. The observed red shift is in good agreement with the decrease in optical band-gap energy obtained from UV–Visible spectroscopy [18].

The visible emission band decreases progressively with increasing annealing temperature, suggesting a reduction in defect-related recombination centers and improved lattice ordering. The suppression of defect emission indicates that thermal treatment effectively reduces the concentration of intrinsic defects within the ZnO lattice. However, for the film annealed at 400 °C, a reduction in the overall PL intensity is observed. This decrease may be attributed to the formation of additional non-radiative recombination centers, defect redistribution, or concentration-quenching effects associated with Eu ion clustering at elevated annealing temperatures [18, 32, 33].

Among all investigated samples, the film annealed at 300 °C exhibits the highest PL intensity, indicating an optimal balance between crystallinity and defect concentration. The enhanced near-band-edge emission and reduced defect-related luminescence demonstrate that annealing at 300 °C is the most favorable condition for improving the optical performance of

Eu-doped ZnO thin films. These results highlight the potential of Eu-doped ZnO nanofilms for ultraviolet-emitting and other optoelectronic device applications.

4. CONCLUSION

10 at.% Eu-doped ZnO nanofilms were deposited on microscope glass substrates by the sol–gel spin-coating technique and subsequently annealed in ambient air at temperatures ranging from 250 to 400 °C for 1 h. The effects of post-annealing temperature on the structural, optical, and photoluminescence properties of the films were systematically investigated. XRD analysis confirmed the formation of a hexagonal wurtzite ZnO structure with a preferred (002) orientation. Annealing improved the crystalline quality of the films and enhanced the diffraction peak intensity. Optical studies revealed high transparency (>90%) in the visible region, while the optical band-gap energy decreased from 3.36 to 3.26 eV with increasing annealing temperature. The absorption intensity was also enhanced after thermal treatment. Photoluminescence measurements exhibited a strong near-band-edge ultraviolet emission and weak defect-related visible emission. The film annealed at 300 °C showed the highest PL intensity, indicating reduced non-radiative recombination and improved optical quality. The results demonstrate that post-annealing effectively tailors the structural, optical and photoluminescence characteristics of Eu-doped ZnO nanofilms, their potential for luminescent, ultraviolet-emitting and optoelectronic device applications.

DECLARATIONS

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Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Availability of data and material

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

Author's contributions

Author's Contribution not available

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