

Synthesis of tin oxide thin films incorporating nanostructured europium oxide via pulsed laser deposition for NO₂ sensing applications

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ABSTRACT

In this work, nanostructured tin oxide (SnO₂) thin films were deposited on silicon substrates at room temperature using pulsed laser deposition (PLD). The study focused on the effect of mixing SnO₂ films with europium oxide (Eu₂O₃) at different weight ratios of 5, 7, and 9 wt% on the gas-sensing properties of the prepared films. X-ray diffraction (XRD) analysis revealed that the addition of Eu₂O₃ contributed to the formation of separate SnO, SnO₂, and Eu₂O₃ phases, which significantly improved nitrogen dioxide (NO₂) sensing performance. Surface morphology analysis showed that particle distribution varied with increasing Eu₂O₃ content. In particular, the sample containing 7 wt% Eu₂O₃ exhibited a narrower particle size distribution and more homogeneous particle growth on the sensor surface, resulting in the highest sensitivity among the prepared samples. The thickness of the deposited films increased with increasing Eu₂O₃ content, although a slight decrease in thickness was observed for the sample containing 5 wt% Eu₂O₃. Gas-sensing results against different NO₂ concentrations showed that the optimum operating temperature was 200 °C. The SnO₂ film mixed with 7 wt% Eu₂O₃ exhibited the highest sensitivity of 626% at 120 ppm NO₂, with a response time of 10 s and a recovery time of 12 s. These findings indicate that Eu₂O₃ mixing can enhance the sensing properties of SnO₂ by modifying its nanostructure and surface morphology, increasing oxygen-vacancy concentration, and improving sensitivity under different environmental conditions.

Keywords: Gas sensing, NO₂, Rare-earth elements, Structural properties, Surface morphology

1 INTRODUCTION

Wide-band-gap semiconducting metal oxides have been studied for several decades and remain an active area of research because of their high stability under ambient conditions, thermal stability, and chemical inertness [1]. Among these metal oxides, tin oxide (SnO₂) is one of the most widely used materials [2]. SnO₂ is an n-type semiconductor with a tetragonal crystal structure [3]. Its electrical conductivity is commonly associated with oxygen vacancies in the crystal lattice [4]. Owing to its wide band gap of approximately 3.6 eV, SnO₂ is a promising material for optoelectronic applications, gas sensors, light-emitting diodes (LEDs), phototransistors,

and solar-cell window layers [5].

However, pure SnO₂ thin films are limited in many device applications because of their relatively low carrier density and mobility. Several approaches have been investigated to improve the physical and chemical properties of SnO₂, including doping with metal ions, such as Sm [6] and Eu [7], or combining it with semiconductors, such as CdO [8], CdS [9], and ZnO [10]. Various techniques have been used to deposit tin oxide nanofilms, including magnetron sputtering [11], sol-gel processing [12], hydrothermal methods [13], and spray pyrolysis [14], to produce nanosized thin-film coatings. Among these methods, pulsed laser deposition (PLD) is a promising technique for depositing nanostructured

thin films of different materials, including metal salts, oxides, and sulfides [15]. PLD is based on directing a high-energy laser beam toward a target material in a vacuum chamber, producing a plasma plume that transfers the target material to the deposition substrate [16]. This technique can produce highly homogeneous thin films with controlled thickness and a relatively fast deposition rate. However, PLD also has limitations, including the need for a controlled vacuum environment, high system cost, and possible restrictions in large-area film uniformity [17].

Doping and/or mixing metal oxides with rare-earth elements is important for improving the electrical, structural, and electronic properties of semiconductors. These modifications can enhance charge-carrier generation and transfer across heterojunctions, thereby improving gas-sensing performance [18]. Rare-earth elements can also enhance surface interactions by increasing the number of active sites, which is crucial for gas-sensing applications [19]. In addition, rare-earth incorporation can modify the optical properties of SnO_2 by introducing new energy levels within the band gap, thereby improving optoelectronic and sensing characteristics [20]. The incorporation of rare-earth elements into catalytic materials may also reduce crystallite size and improve chemical stability [21]. Furthermore, when rare-earth ions occupy substitutional Sn sites in the SnO_2 crystal lattice, they may introduce donor-like behavior, while oxygen vacancies may contribute additional defect-related charge states [22].

Extensive studies have investigated SnO_2 -based sensors prepared using different techniques [23–25]. However, the integration of SnO_2 with europium oxide (Eu_2O_3) via PLD remains an area requiring further investigation. Nanostructured Eu_2O_3 can provide catalytic and electronic effects that may enhance the sensor response to harmful gases, while PLD enables controlled modification of nanostructure and surface morphology in gas-sensing thin films. To address this gap, the present study focuses on the fabrication of hybrid SnO_2 – Eu_2O_3 thin films with different Eu_2O_3 mixing ratios. The structural, morphological, and gas-sensing properties of SnO_2 – Eu_2O_3 films deposited by PLD were systematically investigated.

2 MATERIALS AND METHODS

A Q-switched pulsed Nd laser system (Smart Pico, Deka, Italy), operating at wavelengths of 1064 and 532 nm, was used to deposit unmixed SnO_2 and Eu_2O_3 -mixed SnO_2 thin films. Figure 1 illustrates the schematic

diagram of the PLD system. The deposition chamber consisted of a substrate holder mounted on a rotating base operating at 35 rpm. The vertical distance between the substrate holder and the target was fixed at 4 cm.

A tin oxide disc target, measuring 0.5 cm in thickness and 3 cm in diameter, was prepared from 5 g of SnO_2 powder using a hydraulic press. The resulting target was densified by annealing at 500 °C for 2 h in air. The SnO_2 target was ablated at room temperature, approximately 28 °C, using an Nd laser at a wavelength of 1064 nm, pulse energy of 700 mJ, repetition rate of 6 Hz, pulse duration of 9 ns, energy density of 55.73 mJ/mm² per pulse, and a total of 700 pulses. To improve film purity and laser ablation efficiency, the deposition chamber was evacuated to a pressure of 20 Pa.

Under the same conditions, Eu_2O_3 powder was mixed with SnO_2 at weight percentages of 5, 7, and 9 wt% relative to the total target weight to prepare Eu_2O_3 -mixed SnO_2 targets. N-type silicon substrates with dimensions of 1.5 × 1.5 cm were used for thin-film deposition. The silicon substrates were cleaned by immersion in 99.93% ethanol for 5 min, followed by immersion in a distilled water-hydrofluoric acid solution with a 5:2 ratio for 5 min to remove the native oxide layer. The substrates were then rinsed with distilled water and dried using hot air.

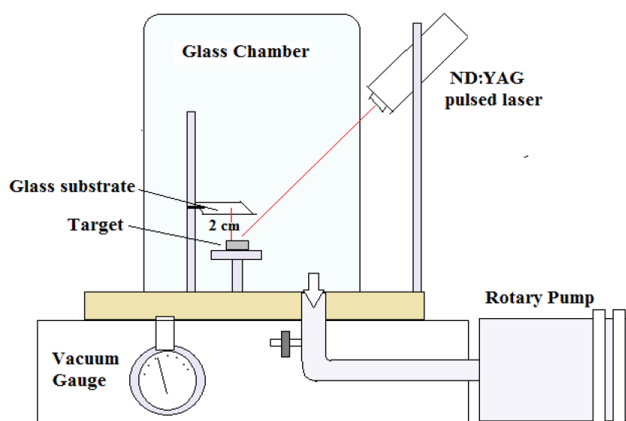


Fig. 1 Scheme of PLD system

The structural characteristics of the deposited films were investigated using X-ray diffraction (XRD; Shimadzu XRD-6000) with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406$). The system was operated at 40 kV and 30 mA over a 2θ range of 10°–80°. Surface morphology was examined using field-emission scanning electron microscopy (FESEM; Inspect F-50).

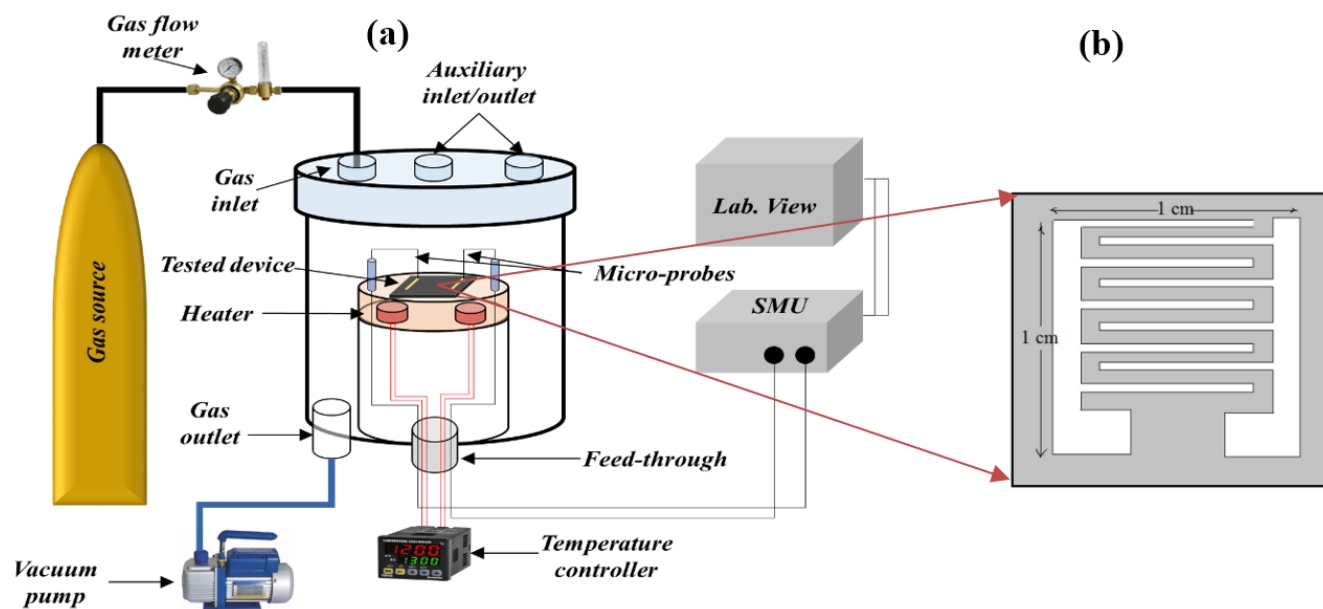


Fig. 2 (a) Schematic diagram of the sensing system (b) Mask used to deposit the gas sensor electrodes

The gas-sensing performance of the films was evaluated using a custom-built gas-testing chamber equipped with electrical measurement tools, as shown in Figure 2a. The films were connected to a two-probe measurement system, and NO_2 gas at controlled concentrations was introduced into the chamber. The chamber was equipped with a gas-flow control system operating within the range of 100-500 sccm. Gas concentrations, expressed in parts per million (ppm), were prepared by diluting the target gas with dry air using volumetric dilution equations. The accuracy of the target gas concentrations was confirmed through preliminary calibration using pre-calibrated gas cylinders.

The mask shown in Figure 2b was designed for the deposition of aluminum electrodes by thermal evaporation for gas-sensor applications. The mask had overall dimensions of 1×1 cm. The interdigitated electrodes formed a comb-like structure consisting of parallel finger-like openings arranged in alternating directions, allowing the formation of two interlocking electrode sets.

Gas-sensing measurements were performed at different operating temperatures of 100, 200, and

300 °C and NO_2 concentrations of 60, 90, and 120 ppm. The sensitivity of the as-deposited and Eu_2O_3 -mixed SnO_2 samples was calculated using Eq. (1) to investigate the effect of Eu_2O_3 content on gas-sensing performance. Changes in electrical resistance were recorded continuously during gas exposure and subsequent gas removal to determine sensitivity, response time, and recovery time.

$$S\% = \frac{R_g - R_a}{R_a} \times 100 \quad (1)$$

where R_a and R_g are the electrical resistance of the sensor recorded in clean air and in the presence of NO_2 , respectively, measured in ohms.

3 RESULTS AND DISCUSSIONS

Figure 3 shows the XRD patterns of the as-deposited SnO_2 and Eu_2O_3 -mixed SnO_2 thin films prepared with different Eu_2O_3 ratios of 5, 7, and 9 wt% using the PLD method. The patterns reveal a polycrystalline structure composed of mixed tetragonal SnO_2 and orthorhombic SnO phases, as identified using JCPDS card Nos. 010-075-9495 and 000-055-0837, respectively.

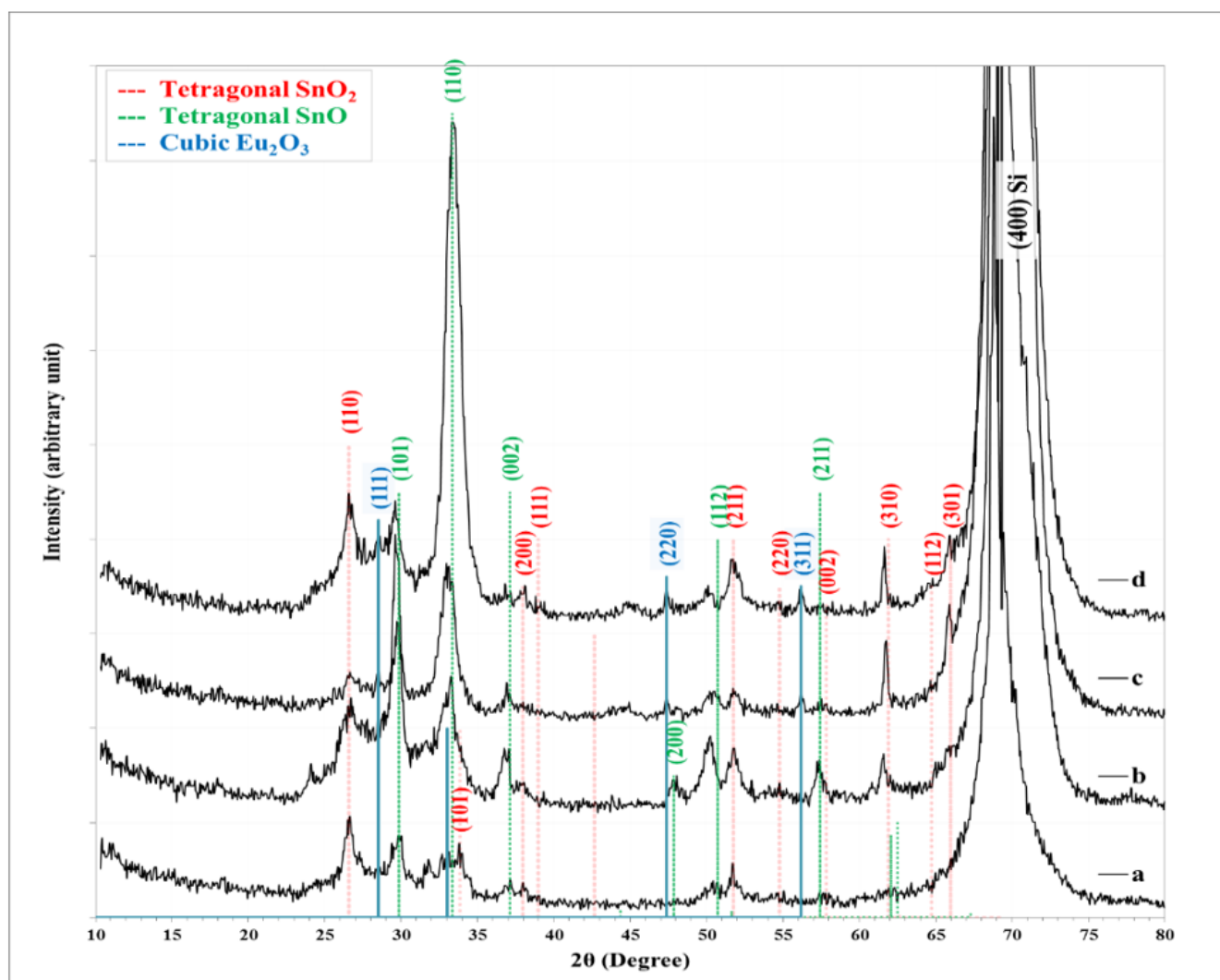


Fig. 3 XRD patterns of SnO₂ and Eu₂O₃-incorporated SnO₂ thin films: (a) 0 wt% Eu₂O₃, (b) 5 wt%, (c) 7 wt%, (d) 9 wt%

Upon mixing Eu₂O₃ with the SnO₂ thin films, the growth of the SnO phase was enhanced more strongly than that of the SnO₂ phase as the Eu₂O₃ mixing ratio increased, indicating a higher degree of oxygen deficiency. With increasing Eu₂O₃ content, new peaks appeared at $2\theta \approx 28.5^\circ$, 47.3° , and 56° , corresponding to cubic Eu₂O₃ according to PDF card No. 00-043-1008. This confirms the formation of a separate Eu₂O₃ phase.

The appearance and coexistence of multiple phases, including SnO₂, SnO, and Eu₂O₃, suggest the formation of nanoscale heterojunctions, which may play an important role in gas sensing. Eu₂O₃ mixing

resulted in a noticeable change in the crystallinity of the SnO and SnO₂ phases, leading to reduced SnO₂ crystallinity and increased dominance of the SnO phase. The incorporation of Eu₂O₃ may also alter the defect structure and oxygen-vacancy concentration of the films, creating localized states within the band gap that can affect the electrical and optical properties [26].

Because the films were deposited by pulsed laser deposition under reduced pressure, the concentration of oxygen vacancies is expected to increase. When atoms from the target material are deposited on the substrate at room temperature, their surface

diffusion length is limited, which can restrict long-range atomic rearrangement and favor polycrystalline phase growth [27]. The interaction between the tin oxide target and the laser-induced plasma plume may also influence the tin-to-oxygen ratio in the deposited film, promoting oxygen-deficient tin oxide phases on the substrate surface [28].

Meanwhile, the possible interaction between europium-containing species and the tin oxide matrix may contribute to the formation of oxygen vacancies in the tin oxide lattice. However, because Eu_2O_3 peaks were observed as a separate phase, substitutional incorporation of Eu into the SnO_2 lattice should be discussed cautiously unless supported by

additional evidence [29].

The crystallite size (D) was determined using the Scherrer equation (Eq. 2) [30], which corresponds to the dominant diffraction peaks of SnO and SnO_2 phases.

$$D = k\lambda/\beta \cos \theta \quad (2)$$

where λ represents the X-ray wavelength, θ denotes the diffraction angle in radians, and k signifies the Scherrer correction factor ($k = 0.89$). Table 1 lists the diffraction angles and crystallite size corresponding to the dominant diffraction peaks of SnO_2 and SnO phases.

Table 1 Diffraction angle and crystallite size corresponding to SnO_2 and SnO structures in thin film deposited at different Eu_2O_3 concentrations

Eu_2O_3 %	2θ (degree)				D (nm)				
	SnO_2		SnO		SnO_2		SnO		Average D (nm)
	(110)	(211)	(101)	(002)	(110)	(211)	(101)	(002)	
0	26.5835	51.7327	29.8727	37.1366	13.10	14.96	12.54	9.13	12.43
5	26.5492	51.8013	29.7014	36.7254	10.82	14.96	10.03	12.16	11.95
7	26.6520	51.8013	29.7699	36.8967	12.45	14.96	13.19	12.77	13
9	26.5520	51.7698	29.5672	36.7282	17.78	12.82	10.90	12.76	13.5

The calculated lattice parameters, as listed in Table 2, show marginal changes in dimensions for both SnO_2 and SnO structures. The Eu_2O_3 contents as a defect induce these variations in lattice parameters.

Table 2 Lattice parameters of SnO_2 structures in thin film deposited at different Eu_2O_3 concentrations

Eu_2O_3 %	SnO_2		SnO	
	a (Å)	c (Å)	a (Å)	c (Å)
0	4.73825	3.18549	3.80041	4.83804
5	4.74426	3.19276	3.80986	4.89031
7	4.72629	3.19880	3.80648	4.86839
9	4.74377	3.17801	3.83731	4.88995

Figure 4 shows the top-view FESEM images at two magnifications, together with the particle-size distributions of the deposited SnO/SnO_2 and Eu_2O_3 -mixed SnO_2 thin films prepared at different Eu_2O_3

mixing ratios of 5, 7, and 9 wt%. The SnO_2 thin film exhibited a continuous surface covered with nearly spherical nanoparticles, with an average particle diameter of 44.53 nm and a standard deviation of 27.1 nm. Mixing Eu_2O_3 with SnO_2 at different ratios significantly influenced particle size and particle-size distribution, thereby affecting the properties of the samples. For the sample containing 5 wt% Eu_2O_3 , the average particle diameter decreased to 29.72 nm, while the standard deviation decreased to 13.27 nm. Increasing the Eu_2O_3 mixing ratio to 7 wt% further reduced the average particle size to 18.62 nm and narrowed the particle-size distribution, with a standard deviation of 7.5 nm. However, increasing the Eu_2O_3 content to 9 wt% increased the average particle size to 38.84 nm and the standard deviation to 15.5 nm.

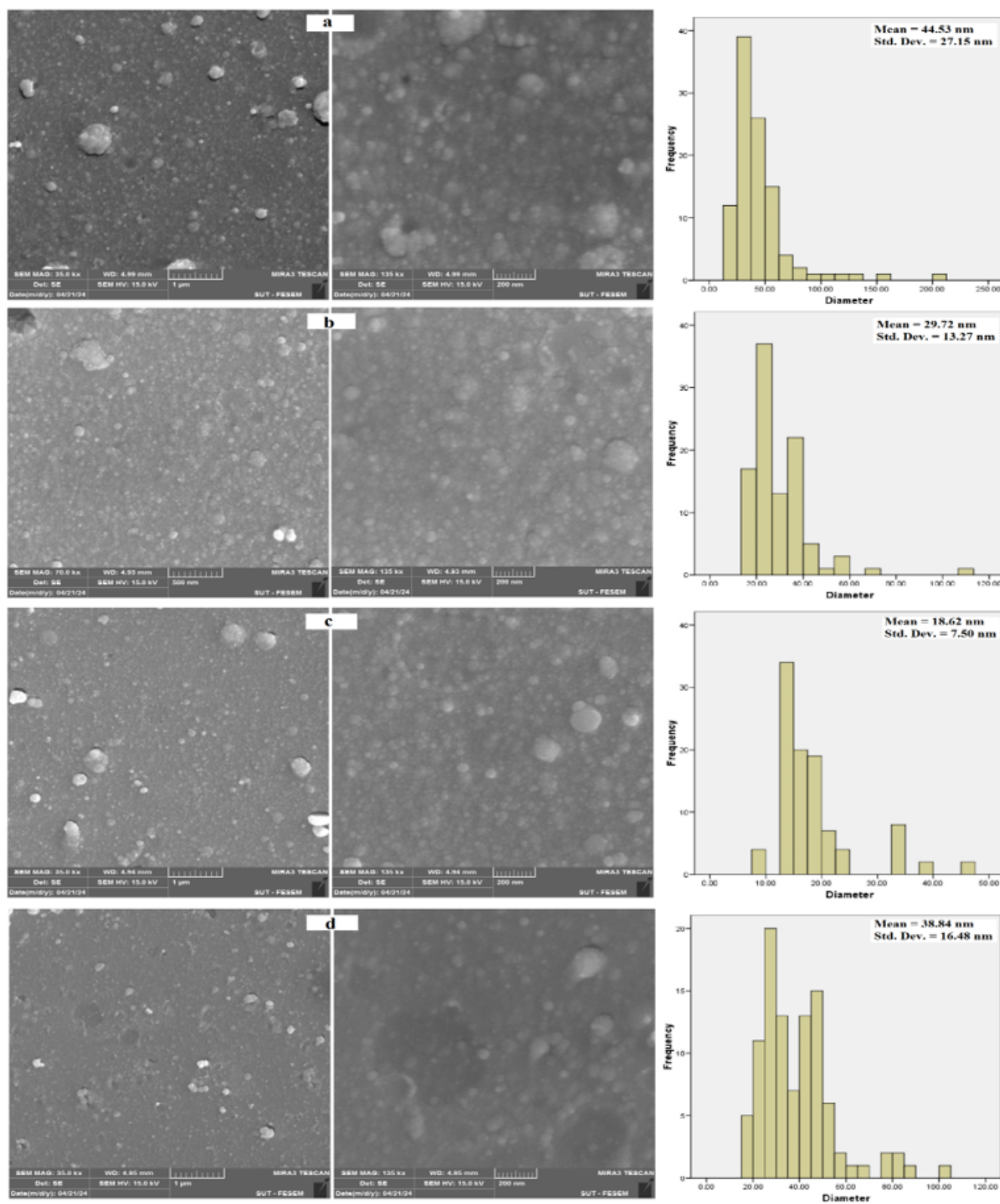


Fig. 4 FE-SEM images and particle size distribution of SnO_2 and Eu_2O_3 -incorporated SnO_2 thin films: (a) 0 wt% Eu_2O_3 , (b) 5wt%, (c) 7wt%, (d) 9wt%

The most uniform particle size and narrowest size distribution were observed at 7wt% Eu_2O_3 , which may contribute to the enhanced gas-sensing performance of this sample.

Figure 5 shows the cross-sectional FESEM images of the as-deposited SnO/SnO_2 and Eu_2O_3 -mixed SnO_2 thin films prepared with 5, 7, and 9 wt% Eu_2O_3 mixing ratios. The deposited films appeared dense and strongly adherent to the substrates, with no visible voids. Increasing the Eu_2O_3 content generally increased the average film thickness from 366.1 nm for the unmixed film to 541.5 nm for the 9 wt% Eu_2O_3 -mixed film, with a slight decrease in thickness observed at 5wt% Eu_2O_3 , reaching 357.2 nm, as listed in Table 3.

The increase in thickness of the Eu_2O_3 -mixed SnO_2 thin films with increasing Eu_2O_3 content may indicate an increased ablation rate of the target material due to changes in thermal properties and energy absorption during laser-matter interaction. In addition, Eu_2O_3 may influence the nucleation and growth processes on the substrate [31,32]. The slight decrease in film thickness at 5wt% Eu_2O_3 may be related to changes in the plasma energy density at this composition, which could reduce the evaporation efficiency of the target material and decrease the amount of material deposited on the substrate surface. This behavior is likely nonlinear, as indicated by the subsequent increase in film thickness at higher Eu_2O_3 contents

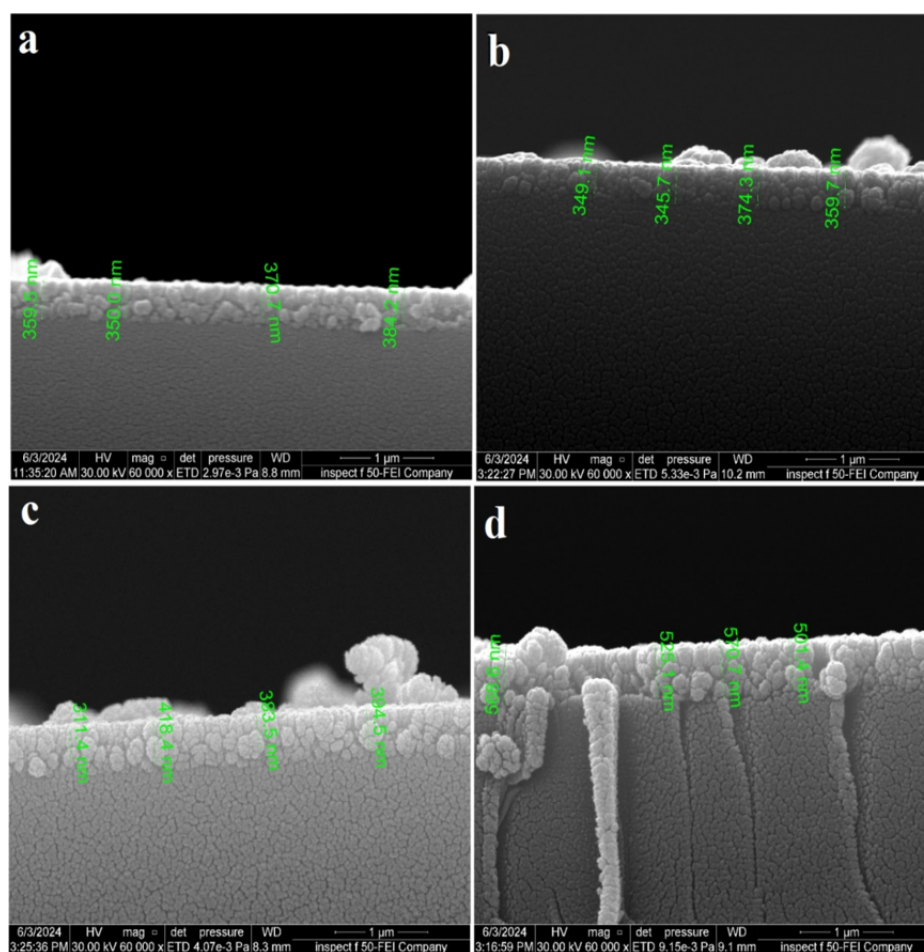


Fig. 5 Cross-sectional FE-SEM images of SnO_2 and Eu_2O_3 -incorporated SnO_2 thin films: (a) 0 wt% Eu_2O_3 , (b) 5 wt%, (c) 7 wt%, (d) 9 wt%

Table 3 Average thickness of the as-deposited and SnO₂ thin film mixed with Eu₂O₃ at different ratios

Sample	Average thickness (nm)
SnO ₂	366.1
5% Eu ₂ O ₃	357.2
7% Eu ₂ O ₃	377.0
9% Eu ₂ O ₃	541.5

Figure 6 illustrates the dynamic response of the as-deposited SnO₂ and Eu₂O₃-mixed SnO₂ sensors containing 5, 7, and 9wt%Eu₂O₃ to 120ppmNO₂ gas at different operating temperatures of 100, 200, and 300°C. The resistance curves show a clear change in resistance during NO₂ exposure and subsequent gas removal, reflecting the response and recovery behavior of the sensors.

The adsorbed oxygen species and the type of oxygen ions formed on the sensor surface depend on the operating temperature; therefore, the resis-

tance variation differs at different temperatures. For all Eu₂O₃ mixing ratios, the electrical resistance increased significantly upon exposure to NO₂, which is consistent with the behavior of n-type semiconductors exposed to an oxidizing gas.

The response and recovery time analysis over the temperature range of 100 – 300°C showed that an operating temperature of 200°C produced the sharpest response, with a response time of 8 s. This behavior may be attributed to enhanced oxygen adsorption and activation at this temperature [33]. The recovery time showed a similar trend, with the pure SnO₂ sensor requiring a longer recovery time than the Eu₂O₃-mixed SnO₂ sensors by approximately 2 s. This indicates that Eu₂O₃ mixing improved the surface reaction kinetics of NO₂ molecules on the sensor surface.

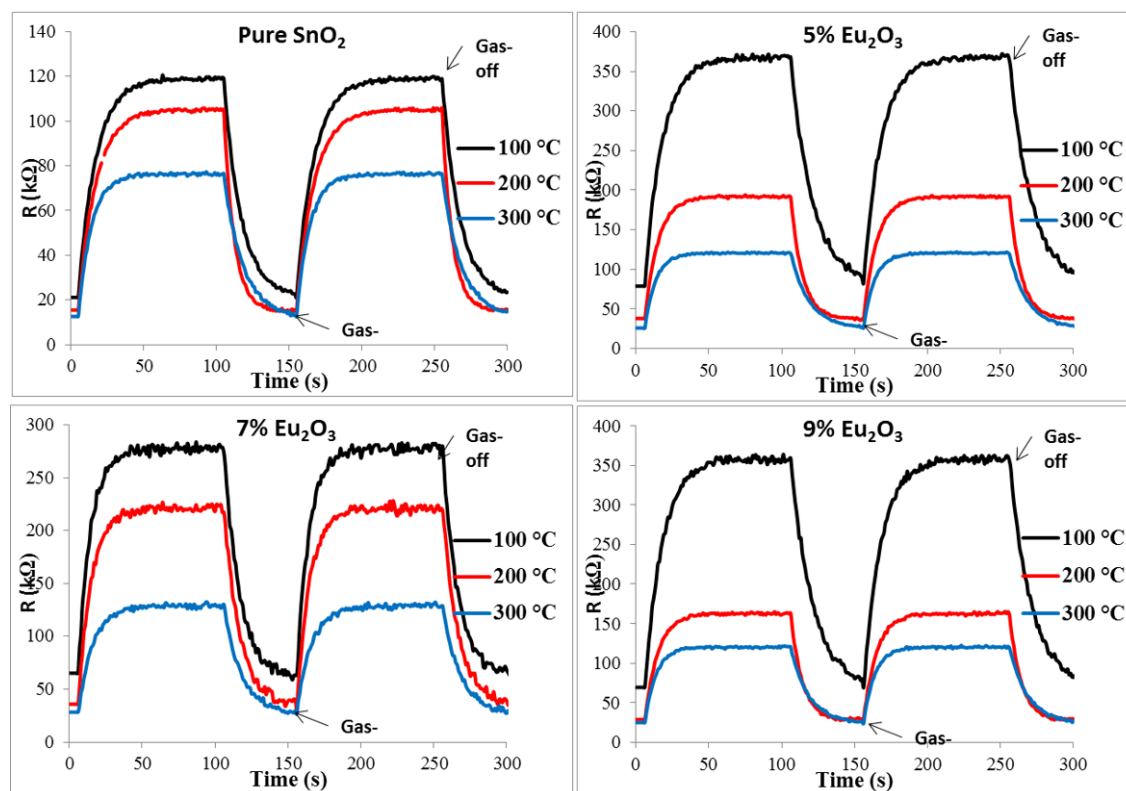
**Fig. 6** Electrical resistance -time plot of the SnO₂ and Eu₂O₃-mixed SnO₂ sensors at different ratios against 120ppmNO₂ at various operating temperatures

Figure 7 shows the resistance variation of the as-deposited SnO_2 and Eu_2O_3 -mixed SnO_2 thin-film sensors at 200°C upon exposure to different NO_2 concentrations of 60, 90, and 120 ppm during gas exposure and gas removal. The sensor resistance increased upon exposure to the oxidizing gas until it reached a stable value, and then decreased exponentially toward its baseline after stopping the NO_2 flow and exposing the sample to clean air. This behavior is typical of n-type semiconductors exposed to oxidizing gases [34].

Oxygen molecules adsorbed on the surface of the active layer extract electrons from the conduction band and form negatively charged oxygen species. When NO_2 molecules interact with the sensor surface, they further capture electrons from the conduction band and/or react with adsorbed oxygen species, increasing the surface depletion layer. As a result, the charge-carrier concentration decreases and the resistance of the sample increases after gas adsorption.

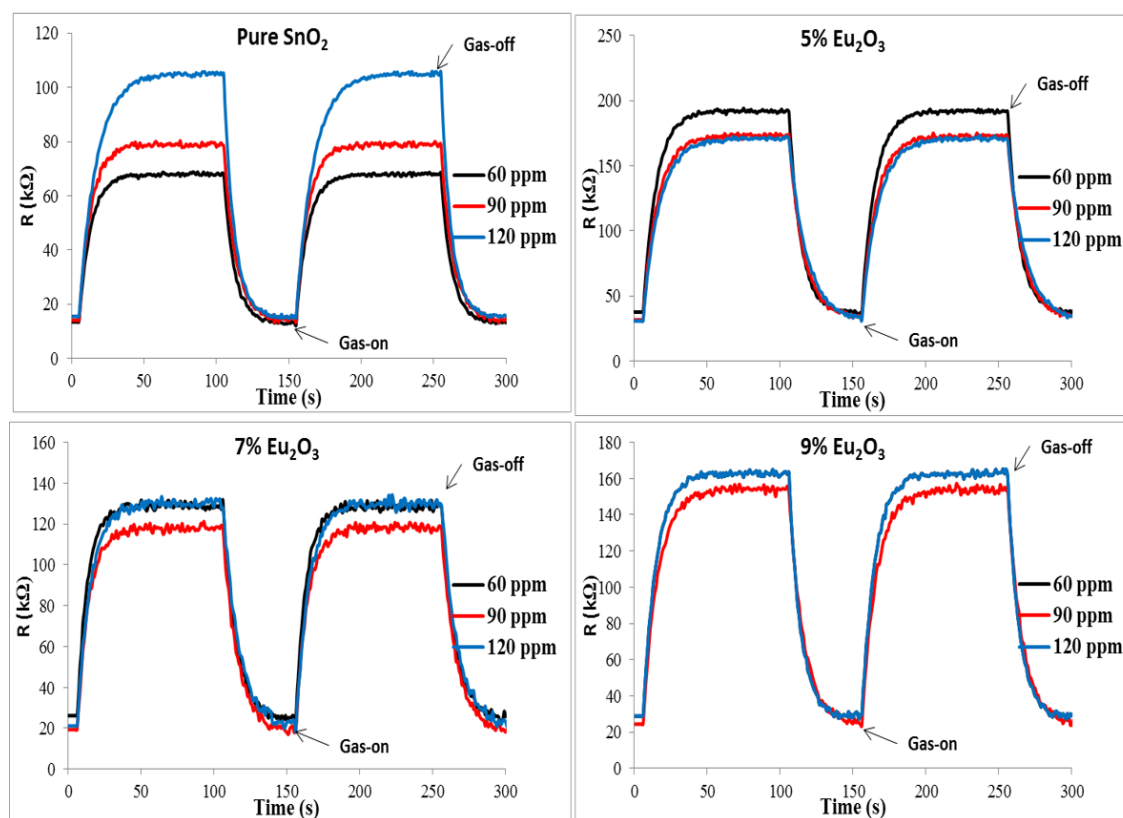


Fig. 7 electrical resistance variation of the Eu_2O_3 -mixed SnO_2 sensors at different ratios at 200°C against (a) 60 ppm, (b) 90 ppm, and (c) 120 ppm NO_2 concentration

Figure 8 shows the sensitivity of SnO_2 and Eu_2O_3 -mixed SnO_2 sensors as a function of operating temperature at a fixed NO_2 concentration of 120 ppm, and as a function of NO_2 concentration at a fixed operating temperature of 200°C . Across all samples, the sensitivity exhibited a temperature-dependent

trend, initially increasing with temperature and then decreasing after reaching a maximum at 200°C . This temperature likely represents an optimal balance between thermal activation of surface reactions and avoidance of rapid gas desorption, thereby maximizing sensitivity. At lower temperatures, the sensitivity

is limited by insufficient activation of adsorbed oxygen species involved in the gas-sensing process; however, this activation improves with increasing temperature up to an optimum limit [35].

Table 4 presents the sensitivity, response time,

and recovery time of the SnO_2 and Eu_2O_3 -mixed SnO_2 sensors at different mixing ratios, as well as their responses to NO_2 gas at different operating temperatures and gas concentrations.

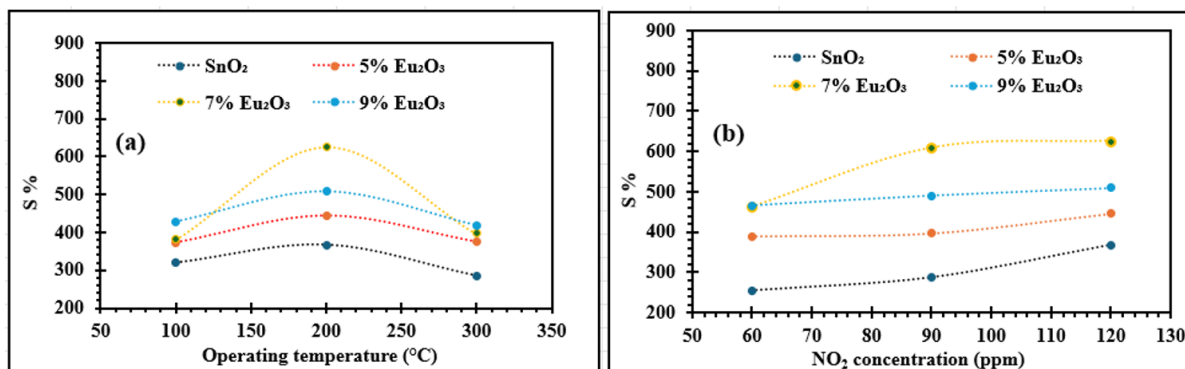


Fig. 8 Sensitivity variation of Eu_2O_3 mixed- SnO_2 sensors at various (a) operating temperatures, (b) NO_2 concentration

Table 4 Sensor parameters of SnO_2 and Eu_2O_3 -mixed SnO_2

T (°C)	NO_2 Con. (ppm)	SnO_2			5 wt% Eu_2O_3			7 wt% Eu_2O_3			9 wt% Eu_2O_3		
		S	Res. Time (s)	Rec. Time (s)	S%	Res. Time (s)	Rec. Time (s)	S%	Res. Time (s)	Rec. Time (s)	S%	Res. Time (s)	Rec. Time (s)
100	120	320	10	12	373	10	12	382	10	12	428	10	12
200	120	367	8	10	445	8	10	626	8	10	509	8	10
300	120	286	10	10	375	10	10	397	10	10	418	10	10
200	60	255	10	12	389	10	12	464	10	12	465	10	12
200	90	287	10	12	396	10	12	609	11	12	490	10	12
200	120	367	10	12	445	10	13	626	12	12	509	10	12

The NO_2 -sensing results indicate that the highest sensitivity was recorded for the 7wt% Eu_2O_3 -mixed sample, with ($S = 626\%$), at the optimum operating temperature of 200°C . This suggests that this mixing ratio was effective in enhancing the interaction between NO_2 molecules and the sensor surface, making the 7wt% Eu_2O_3 -mixed SnO_2 film a promising candidate for gas-sensor applications.

Furthermore, the prepared sensors exhibited sensitivities ranging from 464% to 609% as the NO_2 concentration increased from 60 to 90 ppm. This indicates that increasing the target gas concentration altered the electronic properties of the sensor surface by enhancing gas-surface interactions. The sensitivity further increased to 626% when the NO_2

concentration reached 120 ppm. However, the relatively small increase in sensitivity above 90 ppm may indicate that the sensor surface was approaching saturation. Therefore, increasing the gas concentration beyond this level may not significantly enhance surface interactions or substantially improve sensor sensitivity [36].

4 CONCLUSION

This study demonstrated that mixing SnO_2 with Eu_2O_3 effectively modified the structural and morphological properties of the developed gas sensors. Structural analysis confirmed the formation of separate SnO , Eu_2O_3 , and SnO_2 phases, which may contribute to the generation of oxygen vacancies and

active sites that enhance surface reactions during gas sensing. Surface morphology analysis showed that particle distribution was affected by increasing the Eu_2O_3 mixing ratio.

The 7wt% Eu_2O_3 mixing ratio was the most effective in promoting the interaction between NO_2 gas molecules and the SnO_2 -based sensor surface, resulting in the highest sensitivity of 626% at an optimum operating temperature of 200 °C. Therefore, the 7wt% Eu_2O_3 -mixed SnO_2 sensor can be considered a promising candidate for NO_2 gas-sensing applications.

For future development, coating the surface of Eu_2O_3 -mixed SnO_2 sensors with gold or silver nanoparticles may further improve sensor response. Such noble-metal nanoparticles may enhance electronic transitions and surface catalytic activity at interfaces with different crystalline and morphological structures, thereby improving sensing performance under various environmental conditions.

Authors' contributions

Jamal M. Rzaizj: Proposal, methodology, supervision, validation, and data processing. Writing the original draft. Noor S. Saleh: Software, resources, funding acquisition, investigation, data review, and analysis. All authors contributed to the manuscript text writing.

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Data availability

All the data are presented in this article.

DECLARATIONS

Conflict of interest

The authors declare no competing interests

Consent to publish

N/A

Ethical approval

N/A

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