



The structural, optical, topographical, and H₂ sensing characteristics of a Zn-doped Fe₂O₃ thin layer deposited via DC & RF magnetron co-sputtering method

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ABSTRACT

In this study, a Zn-doped iron oxide layer was deposited onto a microscope slide using the magnetron co-sputtering technique with direct current (DC) and radio frequency (RF) sources. We comprehensively characterized the resulting Zn-doped Fe₂O₃ thin layer, employing techniques such as XRD, Raman spectroscopy, UV–VIS spectrophotometry, SEM, EDX, & AFM. XRD examination showed the nanocrystalline structure in the thin layer under investigation. Based on recorded absorption data, the band gap energy value calculation resulted in a value of 2.23 eV for the thin film. Raman spectroscopy identified peaks possessing Raman shifts from 100 to 1400 cm⁻¹. SEM investigation illustrated a consistently uniform thin film surface characteristic throughout the substrate. Additionally, the AFM study disclosed a small RMS roughness value, indicative of an unrough surface for the Zn: Fe₂O₃ thin layer. The Fe₂O₃ thin film doped with Zn employing a 30 W DC voltage demonstrated effective hydrogen sensing capability at 300 °C, achieving notable response and recovery time. This work presents a novel application of Zn-doped Fe₂O₃ thin films as highly sensitive and stable hydrogen sensors, tailored for high-temperature environments. The unique combination of nanocrystalline structure and Zn doping optimizes the material's electronic properties, enhancing its responsiveness to hydrogen gas. This approach offers a scalable, cost-effective pathway for developing advanced sensor technologies suited to environmental monitoring, industrial safety, and hazardous gas detection, making it a valuable addition to the field of gas-sensing materials.

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1 Introduction

Thin layers composed of iron oxide, including FeO, Fe₃O₄, and Fe₂O₃, are well-recognized, with Fe₂O₃ standing out because of its excellent chemical stability and lack of toxicity. The exceptional electrical, optical, and magnetic characteristics of Fe₂O₃ thin films have led to their widespread use in functional applications such as photocatalysis and capacitive and gas sensing devices [1–3]. Fe₂O₃ exists in two atomic arrangements: α -Fe₂O₃ and γ -Fe₂O₃, which belong to the hexagonal and cubic crystal systems. Additionally, α -Fe₂O₃ is antiferromagnetic, while γ -Fe₂O₃ is ferromagnetic [4]. Compared to γ -Fe₂O₃, α -Fe₂O₃ is steadier and is utilized in many areas more frequently. Currently, methods for preparing α -Fe₂O₃ thin layers include magnetron sputtering [5], hydrothermal technique [6], spraying [7], pulsed laser deposition [8], and sol–gel [9]. These techniques have been extensively utilized to study the sensing features, magnetic properties, and photocatalytic behavior of thin layers made of α -Fe₂O₃ [6–9]. Exploration into α -Fe₂O₃ thin layers using magnetron sputtering has gained significant focus in recent decades. Magnetron sputtering, a form of physical vapor deposition (PVD), allows the deposition of several materials, including alloys, metals, compounds, polymers, oxides, and inorganics [5]. When compared to different methods for depositing films, magnetron sputtering presents benefits such as fast deposition, a compact layer structure, and strong adhesion [10]. Murtaza Bohra and colleagues [11] observed that magnetron-sputtered α -Fe₂O₃ thin films exhibited magnetization approaching the value observed in bulk material and displayed behavior reminiscent of the Verwey transition. As a film with practical applications, understanding the growth pattern and external appearance of α -Fe₂O₃ thin films is crucial for their functional efficiency in applications, for example, photocatalytic performance, magnetism, and mechanical behavior [12, 13]. In the research conducted by Uribe et al. [14], the surface features of α -Fe₂O₃ thin films on inoxidizable steel, produced through RF magnetron sputtering, featured grains arranged in randomly oriented columns; however, a detailed description of its growth mode was not provided. As far as the authors know, few studies have been conducted on the surface properties of α -Fe₂O₃ using magnetron sputtering. Until now, the pattern of growth and surface topography of α -Fe₂O₃ thin

layers produced via magnetron sputtering remain insufficiently understood. This research aims to elucidate the growth mechanism and surface topography of the α -Fe₂O₃ thin layer.

Hematite (α -Fe₂O₃) is an n-type semiconductor used in various applications such as gas sensors, catalysts, magnetic recording media, electrodes, and pigments. This is due to its qualities, which include strong resistance to corrosion, low toxicity, and economical production costs [15, 16]. To introduce p-type characteristics, Mg [17], Zn [18], Cu [19], and N [20] are commonly used as dopants for α -Fe₂O₃. Oxide semiconductors are widely employed in sensors due to their favorable sensing capabilities, including heightened sensitivity, quick response times, and minimal detection thresholds [21]. Despite these advantages, oxide semiconductors face high operating temperatures, limited selectivity, and insufficient reliability. Various strategies, including noble metal doping [22–24], heterojunction formation [22, 24, 25], and methods involving light activation, have been devised to overcome these limitations [26–28]. Notably, forming p–n heterostructures, such as catalyst metal doping [22, 24, 25, 29], is widely acknowledged as a promising approach. The motivation behind this study lies in the need to enhance the hydrogen sensing capabilities of hematite (α -Fe₂O₃) thin films, particularly at high temperatures where gas detection is critical for industrial and environmental safety. Hematite's stability, non-toxicity, and affordability make it a highly attractive material for gas sensing applications, but its intrinsic n-type conductivity limits its performance under specific sensing conditions. Doping α -Fe₂O₃ with Zn introduces p-type characteristics that are expected to improve its sensitivity and selectivity to hydrogen gas, addressing challenges related to response time and reliability. Moreover, the magnetron co-sputtering technique employed in this study allows precise control over the film's composition, microstructure, and surface morphology, which are key factors influencing sensor efficiency. This research aims to provide insights into the structural and surface characteristics of Zn-doped α -Fe₂O₃ thin films, which are relatively unexplored, thereby advancing the development of high-performance, cost-effective H₂ sensors with broad applications in fields requiring reliable gas detection.

This research examines the optical, structural, morphological, elemental, and hydrogen sensing attributes of a thin film of α -Fe₂O₃ modified with Zn. This film

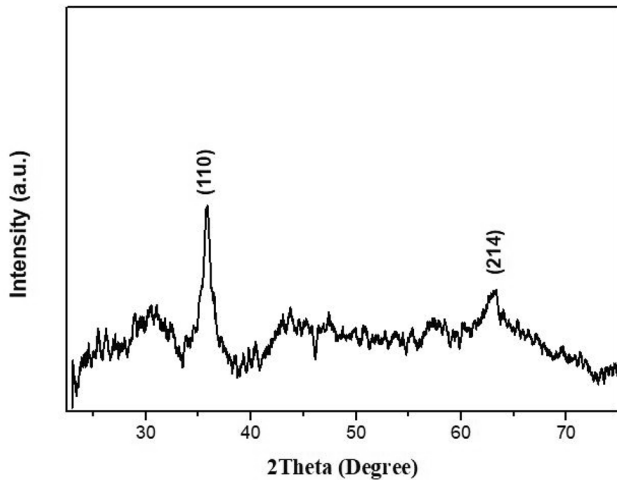


Fig. 1 XRD spectrum of the resulting film

was fabricated via the simultaneous RF and DC magnetron sputtering method.

2 Experimental details

A Zn-doped Fe_2O_3 thin layer was deposited via RF and DC magnetron co-sputtering method at 430 °C substrate temperature with Fe (99.95% purity, Thermo Scientific Alfa Aesar) and Zn (99.99% purity, Alfa Aesar) sputtering targets (2 inches in diameter). RF magnetron sputtering utilized the iron target, while DC magnetron sputtering was employed for the Zn target. The RF and DC power were set to 180 and 30W, respectively. The substrate used was a standard 75 mm × 25 mm × 1 mm glass microscope slide, cleaned by degreasing with an organic solvent, rinsing with water, and drying with nitrogen prior to being placed into the vacuum chamber. The chamber was pumped down to below 4.6×10^{-6} Torr, and argon gas was inserted by a mass flow controller. The target-substrate distance remained fixed at 57 mm. RF and DC sputter guns were positioned downward on the top plate, while the microscope slide rested on a heater at the base plate of the sputter chamber. Throughout the film deposition process, the working pressure, argon flow rate, and oxygen flow rate were maintained at 7.6×10^{-3} Torr, 42 sccm, and 2 sccm respectively. The substrate was continuously spun during the process to ensure uniform film deposition. The rotational speed of the substrate was set at 3 revolutions per minute (rpm). The film deposition lasted for 35 min.

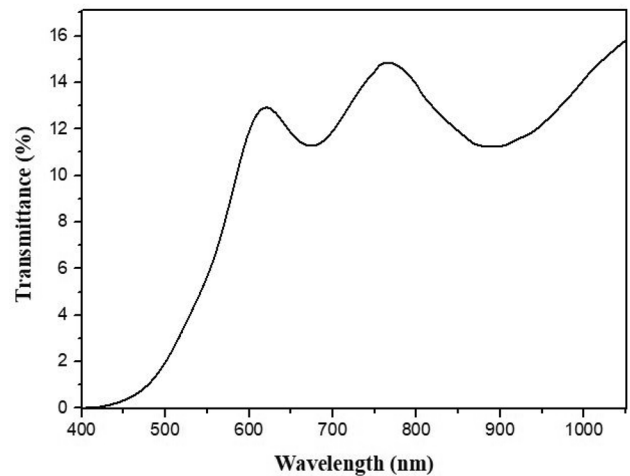


Fig. 2 Optical transmittance spectrum of the generated thin film

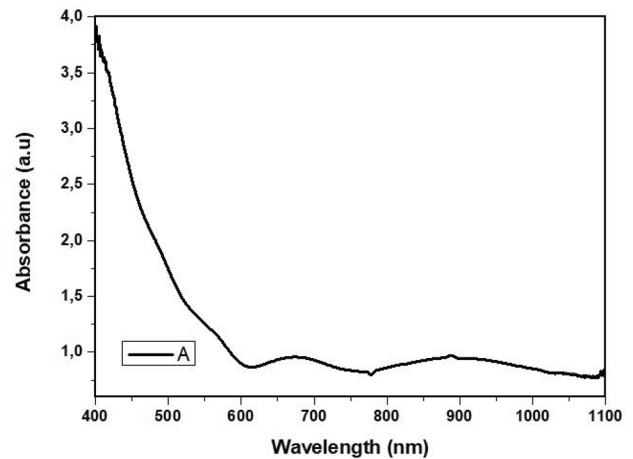


Fig. 3 The spectrum of optical absorption of the generated thin film

The resulting Zn-doped Fe_2O_3 thin film was examined using Raman micro-spectroscopy, XRD, SEM, AFM, and XPS. Using the Specs-Flex brand system, XPS measurement was carried out at an X-ray energy of 1486.6 electron volts. An aluminum anode serving as the source of X-rays was run at 15 kV and 400 W for the measurement. A PANalytical Empyrean system with a $\text{CuK}\alpha 1$ source was used for XRD measurement to examine the thin-film structure. Optical phonon mode analysis was performed using a Witec Alpha 300R Raman spectrometer. The optical characterization was carried out by a LAMBDA 1050 UV/VIS/NIR spectrophotometer. The Zeiss Sigma300 model SEM was employed for surface and morphological

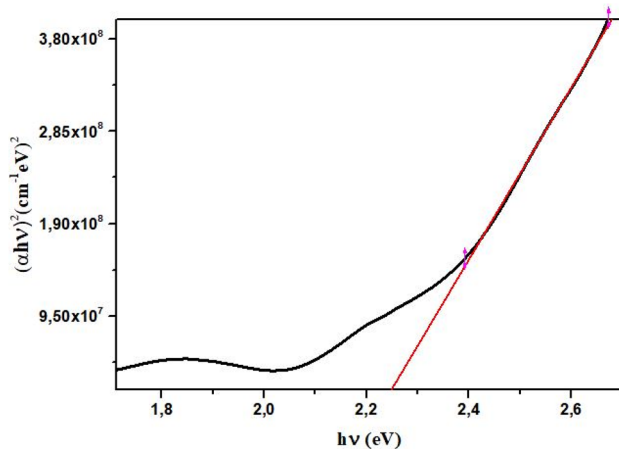


Fig. 4 Tauc plot comparing $(\alpha h\nu)^2$ against $h\nu$

examination. Using the AFM-500II, the thin film surface topography was examined. Al metal spraying is the most appropriate method for producing comb-shaped interdigitate electrodes (IDT), which can yield more sensitive and accurate results for gas sensor measurements. The surface area of the measured thin film is $1.2 \text{ cm} \times 1.2 \text{ cm}$, and the IDT contact distance between the electrodes is $500 \text{ }\mu\text{m}$. The sensor metal Al was evaporated onto the sample using the thermal evaporation technique. Before performing gas detection measurements, 500 sccm of dry air and H_2 gas with different standard sccms were combined to reach the required H_2 parts per million (ppm) levels of 1000 ppm at $300 \text{ }^\circ\text{C}$. Before beginning the measurement, the system's interior was appropriately cleaned. To ensure accurate results, a complete cycle of sensor measurement was initiated under dry atmospheric conditions. Next, 1000 ppm dry air + H_2 gas was sent to the gas sensor test room, where the electric current generated by the voltage source and charge carriers was measured at a constant voltage of 2 V in this different gas environment.

3 Results and discussion

The X-ray diffraction analysis result of the deposited Fe_2O_3 thin film within the 2θ angle range of 20° to 75° is displayed in Fig. 1. According to the (JCPDS Card No. 33-0664), the prominent 2θ peak patterns at approximately 36° and 63° correspond to the (110) and (214) The planes representative of the

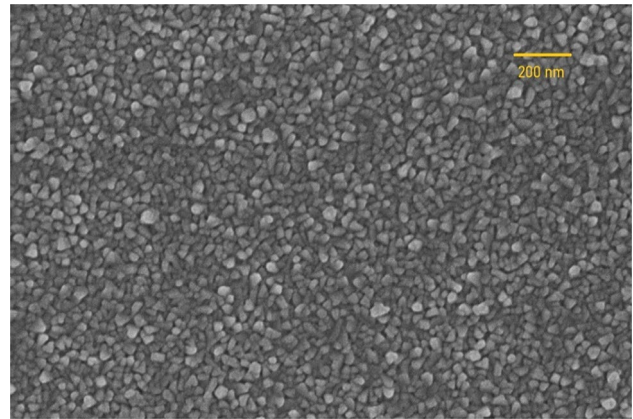


Fig. 5 SEM image magnified to 50,000 times the resulting film

typical hematite phase of Fe_2O_3 , featuring structural dimensions where both a and b measure $5.034 \text{ }\text{\AA}$ and c measures $13.721 \text{ }\text{\AA}$ [30–34].

Scherrer's relation was applied to determine the approximate crystallite size in the thin film based on half the width of the main diffraction peak [35, 36]:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where λ is the wavelength ($1.5406 \text{ }\text{\AA}$) of X-ray, β is the full width at half maximum (FWHM) of the sharp peak in the pattern (in radians), θ is the angle of diffraction, and D is the crystallite diameter. A mean crystallite size of 17 nm suggests that the layer's structure is nanocrystalline. The micro-strain value can be calculated as follows [37]:

$$\varepsilon_X = \varepsilon_{\max} \exp \left(-c \left(\frac{D/2}{x} - 1 \right) \right) \quad (2)$$

where c is a constant, it characterizes the increase in strain from the center of the sphere to its surface and is referred to as the strain exponent. The microstrain value for $2\theta = 36^\circ$ is approximately 0.0143, and for $2\theta = 63^\circ$ approximately 0.0269, respectively.

Since x_f^* represents the displacement within the nanoparticle where the number of d-planes is reduced by half, it is not a fixed value and varies based on the broadening w , as described by the following relationship:

$$X_f^* = \frac{k}{w_{av}} \quad (3)$$

where k is constant.

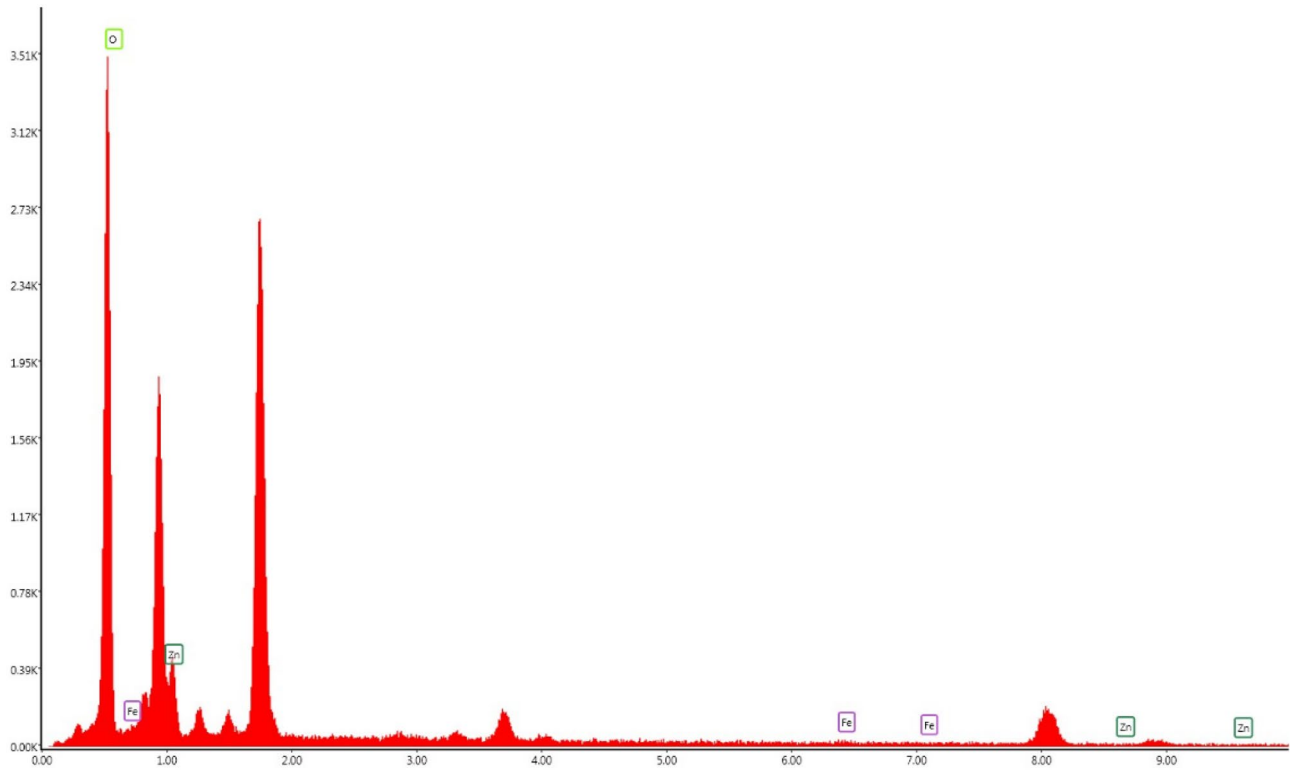


Fig. 6 EDX spectrum of the deposited Zn-doped Fe₂O₃ thin film

$$X_f = 0.62D/c \quad (4)$$

The reciprocal of the position from the center of the nanoparticle at FWHM is 77.5 nm.

The crystalline size can be calculated by the following [37]:

$$w(2 \cot \theta_0 - w) = \left(1.6 \times 10^{-2}/D\right) \left(\frac{d_0}{w}\right) - \left(\frac{6.2 \times 10^{-3}}{c}\right) \quad (5)$$

The crystallite size of 17 nm observed in this study is smaller than the reported value of approximately 30 nm for Fe₂O₃ obtained using the strain distribution (SD) model, in the literature [37].

The produced film's optical transmission and absorption were examined in the 400–1100 nm wavelength range. Figures 2 and 3 display the acquired transmittance and absorbance spectra. Based on the figure, the transmittance curves' oscillatory nature can be attributed to the interference effect that results from multiple reflections at the interfaces. The transmittance spectra and the absorbance spectrum exhibit precisely the opposite trend. Experiments on the optical absorption of materials are crucial for understanding optical

transitions that can be induced to study critical aspects of the band structure and energy gap. The energy band gap (E_g) is calculated from absorption data through the Tauc equation [38]:

$$\alpha h\nu = A(h\nu - E_g)^n \quad (6)$$

where A is an energy-dependent constant of thin film, $h\nu$ is the incident photon's energy, and α is the absorption coefficient. The energy gap E_g takes values 1/2, 3/2, 2, 3 depending on the electronic transition [39–41] Fig. 4 illustrates the $(\alpha h\nu)^2$ versus $h\nu$ plot for Zn-doped α -Fe₂O₃ nanoparticles, demonstrating their direct band gap properties [42]. The following relation can be used to determine the absorption coefficient α signifies the rate at which light intensity decreases exponentially as it penetrates the solid material [43, 44].

$$\alpha = 2.303(A/t) \quad (7)$$

where the thickness of the film is t . Figure 4 shows the Tauc plot of $(\alpha h\nu)^2$ about the photon energy $h\nu$. The material's band gap energy is shown in the figure as

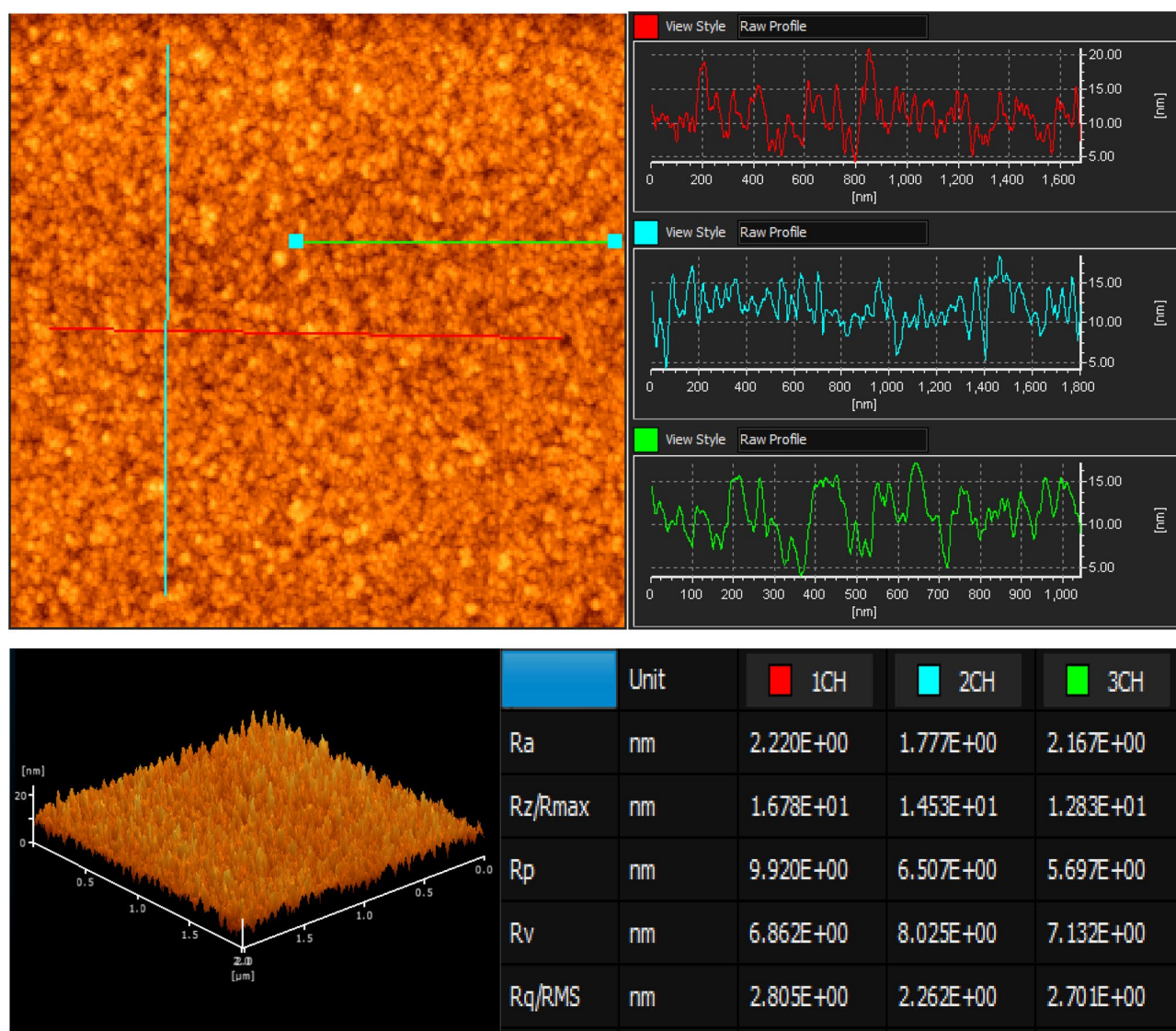


Fig. 7 2D and 3D AFM image of the resulting film

the value of the horizontal axis' intersection point. A direct band gap is present in the film, as indicated by the straight-line behavior. The inferred band gap of the film, at 2.23 eV, closely aligns with the established energy gap of Fe_2O_3 .

The SEM image at 50,000 magnifications corresponding to the surface morphology of the thin layer deposited onto the glass substrate is displayed in Fig. 5. The image indicates that the film possesses a crystalline structure with uniformly sized crystals.

Based on SEM characterization, the average size of the crystallites is approximately 40 nm. The continuous, homogeneous, dense film surface is visible and devoid of holes or cracks. The Zn-doped Fe_2O_3 sample was subjected to an elemental analysis using Energy-Dispersive X-ray analysis. Figure 6 demonstrates that the prepared sample comprises iron, oxygen, and zinc elements. The chemical composition of the thin film was verified using energy-dispersive X-ray (EDX) analysis, as shown in Fig. 6. The spectrum reveals the atomic percentages (at%) of iron, oxygen, and zinc in the sample to be 38.9%, 60.5%, and 0.6% respectively.

The AFM analysis in Fig. 7 illustrates the fabricated thin film's 2D and 3D surface topography. AFM

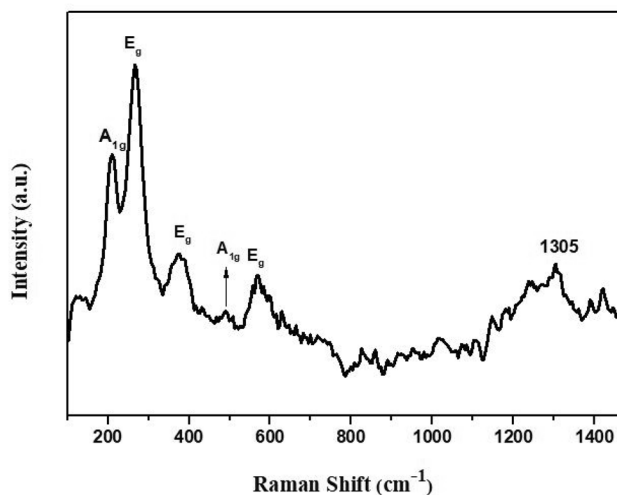


Fig. 8 Raman spectrum of the produced film

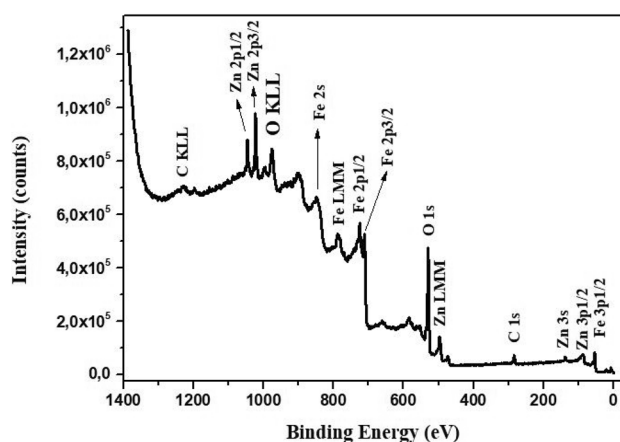


Fig. 9 XPS survey of the produced thin film

images depict uniform, continuous, and densely packed nanostructures composed of columnar grains. The tallest features reach approximately 20 nm, with the most profound areas measuring around 5 nm. The value of average roughness, represented by R_q /RMS, is approximately 2.6 nm.

Raman spectroscopy is an effective technique for distinguishing and distinguishing oxides. The Raman spectrum of the deposited Zn: Fe_2O_3 thin film is given in Fig. 8. The hematite thin layer displays strong Raman peaks at 218 cm^{-1} , 245 cm^{-1} , 370 cm^{-1} , 496 cm^{-1} , 570 cm^{-1} , and 1305 cm^{-1} , as observed in Fig. 8. Hematite belongs to the D_{3d} point group, which determines the specific Raman-active modes observed. The modes that are active in Raman spectroscopy A_{1g} (1) and A_{1g} (2) correspond to the peaks at 218 cm^{-1} and 496 cm^{-1} , while the E_g Raman-active modes are linked to the peaks at 245 cm^{-1} , 370 cm^{-1} and 570 cm^{-1} [45]. The 281 cm^{-1} and 400 cm^{-1} peaks are associated with the stretching (Fe–O) mode between two Fe and O atoms [46]. The Raman peak at 570 cm^{-1} is credited to the longitudinal optical (LO) mode of hematite. The strong peak at 1305 cm^{-1} can be linked to two-magnon scattering, arise from the interaction of two magnons in nearly antiparallel spin states [47].

XPS spectrum was utilized to examine the electronic characteristics of Zn-doped Fe_2O_3 thin film. The spectrum (Fig. 9) revealed the presence of Fe, O, C, and Zn in the film. Prominent signals of Fe 2p $_{3/2}$, C 1s, and O 1s were observed. Peaks at 713.12 eV and 727.17 eV correspond to Fe 2p $_{3/2}$ and Fe 2p $_{1/2}$, respectively. The Fe 2p $_{3/2}$ peak was noticeably sharper and more intense than the Fe 2p $_{1/2}$ peak, attributed to

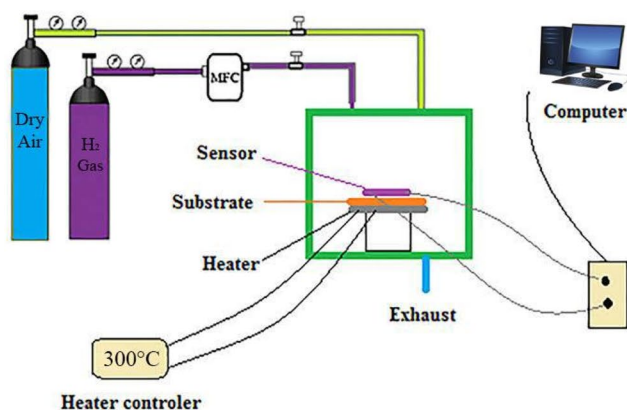
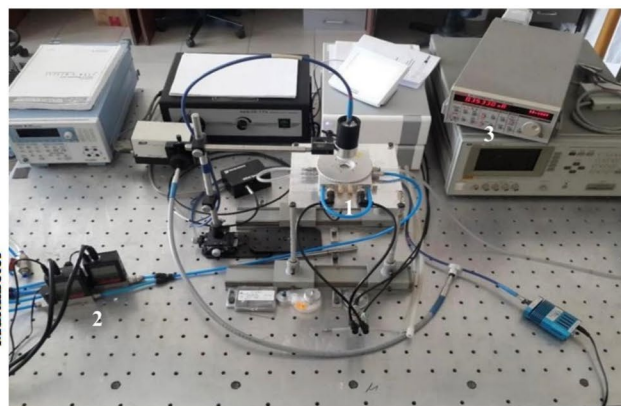


Fig. 10 **a** Schematic of the gas sensor measurement system **b** picture of the gas sensor measurement system (1) Gas sensor testing room (2) Gas flow control unit (3) Amperometer

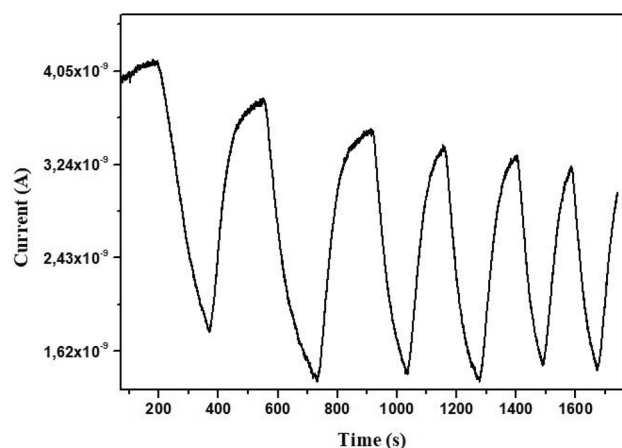


Fig. 11 Gas sensors response of the Zn-doped Fe_2O_3 thin film for 1000 ppm at 300 °C

the interaction between the spin and orbital motion [48]. The energy difference of 14.05 eV between Fe 2p_{3/2} and Fe 2p_{1/2} aligns with literature values [49]. These peaks are typical of the Fe^{3+} state in Fe_2O_3 [50]. A strong C 1 s peak at 287 eV, resulting from C–C bonds, was due to carbon contamination from the air [51]. The O 1 s spectrum peaked at 530 eV, representing the O atoms in $\alpha\text{-Fe}_2\text{O}_3$ (Figs. 9 and 10).

The schematic and picture of the gas sensor measurement system are shown in Fig. 10a, b, respectively. When oxygen gas on the film surface interacts with hydrogen (in dry air mixed with H_2), the free electrons from the oxygen impart a negative charge at the band edge and combine with the film, which is a p-type semiconductor material. This leads to a reduction in current due to electron–hole recombination, thereby decreasing the material’s conductivity (as illustrated in Fig. 11). The working principle of gas sensors relies on changes in conductance when the sensing material is subjected to target gases. These changes in conductivity arise from the adsorption of gas molecules and the exchange of electrons among particles in the reactive state on the semiconductor surface. The gas sensor response is estimated using the equation $\%R = [(I_0 - I)/I_0] \times 100$, where R represents the sensor response, I_0 is the initial current, and I is the final current. Measurement was conducted at 300 °C with 1000 ppm H_2 gas. Fe_2O_3 thin film exhibits an approximately 60% response to 1000 ppm H_2 gas at 300 °C.

4 Conclusion

This research successfully synthesized a thin film of Zn-doped Fe_2O_3 via the RF and DC magnetron co-sputtering method. The investigation focused on understanding the optical, structural, and morphological characteristics concerning the dopant concentration controlled by the power applied to the Zn target. XRD study indicated a nanocrystalline structure in the Zn-doped $\alpha\text{-Fe}_2\text{O}_3$ thin film, while Raman analysis verified the presence of a hematite structure. The energy bandgap of the hematite thin film was determined to be 2.23 eV. The surface topography of Zn-doped Fe_2O_3 thin films played a crucial role in enhancing the gas sensor’s sensitivity, resulting in a remarkably rapid response and recovery time. Furthermore, the material’s p-type conductivity, enabled by Zn^{2+} cationic impurities, opens the potential for its use in photocatalytic and electrochemical applications. This study presents a method for developing straightforward and cost-effective designs for H_2 sensors using Zn-doped Fe_2O_3 thin film. Fe_2O_3 thin films doped with various elements can improve performance in gas sensors and photocatalytic devices, making the Zn-doped Fe_2O_3 thin film a strong candidate for multifunctional sensor technologies. Consequently, this study provides a scalable, cost-effective approach to designing H_2 sensors using Zn-doped Fe_2O_3 thin films, with broad applications in environmental sensing and energy systems.

Author contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Günay MERHAN MUĞLU, Volkan ŞENAY, Maryam ABDOLAHPOUR SALARI, Sevda SARITAŞ and Mutlu KUNDAKÇI. All authors read and approved the final manuscript.

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Data availability

The datasets generated during and/or analysed during the current study are available from the authors.

Declarations

Competing interests The authors have no competing interests to declare that are relevant to the content of this article.

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