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Study the Optical Properties of Al-doping in ZnO Thin Film Grown by Sol-gel Spin Coating Technique

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Abstract

The study explored the effects of aluminum doping on the optical and electrical properties of ZnO thin films, along with their gas-sensing capabilities, specifically in response to blood serum. ZnO thin films were prepared using a spin-coating method, followed by annealing at 400°C, with varying Al doping concentrations (0%, 1%, 2%, 3%, 4%, and 5%). In this work, we investigate the influence of Al doping on ZnO to detect various concentrations of acetone vapor. It was found that the optical band gap of Al-ZnO varies from 3.045 eV to 3.385 eV, and the aluminium concentration in ZnO films increased from 0% to 5%. Optical properties have been studied, and the observed band gap lies in the range of (3.045 - 3.385) eV. The 1% Al- ZnO sample showed a better response than the undoped ZnO film. The increase in the percentage of dopant i.e., Aluminium in the ZnO thin film, the sensitivity was somewhat increased. Time-dependent drift in resistance of ZnO films has been observed. ZnO films showed a fast and slow resistance change response when exposed to gases with varying volumes. The maximum values of sensitivity in terms of resistance were found to be 199.20 for doped and 189 for Al-doped and undoped ZnO thin film for 3 ml volume. The decreased resistivity with increasing Al concentration may be attributed to an increase in both carrier concentration and Hall mobility.

Introduction

A thin film is generally defined as a film having a very thin layer up to a thickness of 10 nm to 1-2 µm. A thin film is a thin layer that is deposited on the supporting material known as substrate. The films can be classified based on the material into various categories such as metallic, dielectric, organic, etc. in which the properties of the film can be altered or modulated using various techniques like doping, thickness variation, or surface treatment (Marasini *et al.*, 2024). Zinc Oxide has many physical and chemical properties by which it is used in various fields as well as for preparing sensors for the detection of toxic chemicals, gases, or biomolecules. ZnO-based gas sensors are broadly employed in various applications due to their excellent sensing response, good selectivity, easy fabrication, low cost, good thermal and chemical stability, and non-toxicity (Chopra *et al.*, 2004). ZnO is enormously growing because of its excellent optical, electrical, magnetic, piezoelectric, catalytic, and gas-sensing properties that make it specifically attractive for nanoelectronics, optoelectronic, nanophotonic, and piezoelectric devices (Rai *et* *al.*, 2012). Different nanostructures of ZnO including nanorods, nanowires, nanotubes, and nanoribbons. The processing temperature of ZnO nanostructures is very low. Therefore, cheap substrates like glass and plastic can also be used for fabricating ZnO-based devices. Moreover, the electrical and optical properties of ZnO can be easily tuned by post-deposition treatments like annealing, surface treatments, and doping with materials like Aluminium, gallium, indium, tin, and copper. It is an n-type transparent material. It can also be used for near-UV emission and detection, as a transparent conductor, and as a channel material in TFT (Ozgur *et al.*, 2010). ZnO is an intrinsically n-type semiconductor having a band gap of 3.37 eV and a large exciton binding energy of 60 meV (Park *et al.*, 2006).

As ZnO is a chemo-resistive sensor, the change in its resistance is highly dependent on the presence of chemisorbed oxygen ions. In addition, oxygen molecules are absorbed on the surface of ZnO in the presence of atmospheric air. Their formation thus occurs due to the extraction of electrons from the conduction band of ZnO which increases the resistance of ZnO. When reducing gases interact with the chemisorbed oxygen ions on the surface of ZnO, the reduction in resistance takes place because oxygen ions donate free electrons to the conduction band of ZnO. Furthermore, the sensing response of a ZnO-based gas sensor is significantly affected by the working temperature and gas concentration (Kumar *et al.*, 2017).

The ZnO thin film can be deposited by using various deposition techniques, such as magnetron sputtering, pulsed laser deposition, chemical vapor deposition, spray pyrolysis, and sol-gel method. ZnO thin film applications are widely used today, and these materials have been a significant concern in both basic and applied sciences. In this study, sol-gel spin coating techniques were used to create zinc oxide (ZnO) nanoparticles. According to research on optical properties, the observed band gap is between 3.32 and 3.36 eV (Gul, 2012).

ZnO as a Gas sensor

Generally, gas sensors known as gas detectors are electronic devices that detect and identify or respond to different types of gases (Shrestha *et al.*, 2010). A device that detects the presence or concentration of gases in the atmosphere is called a gas sensor. By altering the resistance of the material inside the sensor, the sensor generates a corresponding potential difference based on the gas concentration, which may be recorded as output voltage. The type and concentration of the gas can be inferred from this voltage value (Toledo, 2022). The gas sensing mechanism of ZnO nanostructures-based gas sensors relies on the alteration of a depletion layer on the surface of ZnO nanostructures. The oxygen molecules are absorbed on the ZnO surface in the presence of atmospheric air. Furthermore, the formation of oxygen ions occurs on the surface due to the extraction of electrons from the conduction band of ZnO, leading to an increase in the resistance of ZnO (Gas Sensors, 2022).

In the sensing to improve electrical and optical properties, ZnO film can be doped with many metal elements such as Aluminium, Indium, Gallium, and Barium affected to high electrical conductivity (Den, 2018). Among these elements, Al-doped ZnO is considered an alternative for transparent conducting oxide material (TCO) for solar cells and light-emitting diodes (LEDs) (Dunes *et al.*, 2002).

The atomic substitution of Al to Zn in the ZnO crystal structure has occurred in a free electron in the conduction band, which provides a reduction in its electrical resistivity (Shahedi and Jafari, 2016). Al-doped ZnO shows high transparency and low resistivity (Maldonado & Stashans, 2010). The AZO thin films have been prepared by various methods such as sol-gel processing (Raghu *et al.*, 2017), also doping method known as magnetron sputtering (Zhou *et al.*, 2007), and atomic layer deposition can be used which are easy and convenient ways of doping (Wang and Zhang, 2017). Al-doping of ZnO aerogel leads to the improvement of the crystalline quality due to the occupation of Zn^{2+} sites by Al^{3+} ions (Zhai *et al.*, 2016). Al-doping increased the band gap energy of ZnO nanoparticles. Al-doping also enhanced the cytotoxicity and oxidative stress response of ZnO nanoparticles in MCF-7 cells (Fatah *et al.,* 2020).

Photoluminescence is when light energy, or photons, stimulate the emission of a photon. Zinc oxide has a wide variety of applications as a luminescent material, such as vacuum fluorescent displays due to its room-temperature ultraviolet emission and nonlinear optical properties. The combination of being a large-bandgap semiconductor and a luminescence material has allowed us to study nanostructured ZnO such as nanoparticles, nanowires, nanobelts, and nanotubes (Akhtar *et al.,* 2015). As photoconductors in electrophotography, ceramic varistors, and sensor elements in the detection of flammable gases, ZnO compounds doped with impurities are utilized. The piezoelectric characteristics of ZnO as a thin film make it suitable for use in pressure transducers and various acoustic-optical systems. Because of its non-linear optical characteristics, ZnO doped with Al, and I is utilized to make transparent conductive electrodes for fluorescent screens (Bhatai *et al.*, 2017).

Measuring refractive index *n* is essential for the optical characterization of optical devices such as optical switches, filters, modulation, and other optoelectronic devices. The refractive index *n* of thin films can be calculated from the formula, $n = (1 + R/1 - R) + (4R/(1 - R)2) - k2$. The resultant n is a function of incident light wavelength. Measuring the complex refractive index ($N = n + ik$) is crucial for the characterization of the optical properties of thin films (Liu *et al.*, 2002).

The experimental value of the refractive index of the ZnO thin film is calculated using the fringe envelop method. The formula used to calculate the refractive index is given in the equation below.

$$n = \sqrt{N + \sqrt{N^2 + n_s^2}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots.(i)$$

Where $N = \left(\frac{1.5^2+1}{2}\right) + \left(3 \times \frac{(T_M - T_m)}{(T_M \times T_m)}\right)$

T_M and T_m are maximum and minimum transmission respectively, on the envelope at a certain λ, and refractive index ns of the substrate. The envelopes connecting the interference maxima and minima are continuous functions of the wavelength λ. Therefore, for each maximum of the transmission curves (T_M) corresponding minimum (T_m) may be determined at the same λ, and vice versa. The accurate measurement of the imaginary part of the complex refractive index, called extinction coefficient (k) is important for optical applications based on the absorption of light. To investigate the extinction coefficient (k), the average thickness (d) of films should be determined. The extinction coefficient (k) was calculated easily by the simple formula; $k = \alpha\lambda/4\pi$, where α is the absorption coefficient defined by; $\alpha = (1/d) \ln(1/T)$ (Valle *et al.*, 2004).

Materials and Methods

Preparation and cleaning of a glass substrate

Glass substrates were cut into 2cm x2cm pieces, cleaned with dilute Hydrochloric Acid (HCl), boiled in acetone at $80^{o}C$, and then dried. The boiled glass substrate was washed and cleaned with alcohol (Ethyl alcohol). The cleaned glass substrate was kept in a free environment.

Preparation of Zinc acetate solution

The mother solution for samples was prepared by dissolving an appropriate amount of Zn acetate dihydrate. The choice of this as a source solution is because hydrolysis of the acetate group gives the product that is soluble in the solvent medium (Marasini *et al.*, 2024). A solution of 0.5 molalities was required for the preparation of the solution, so 5.478-gram Zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) was measured with the help of an electrical weighting machine as shown in the figure below.

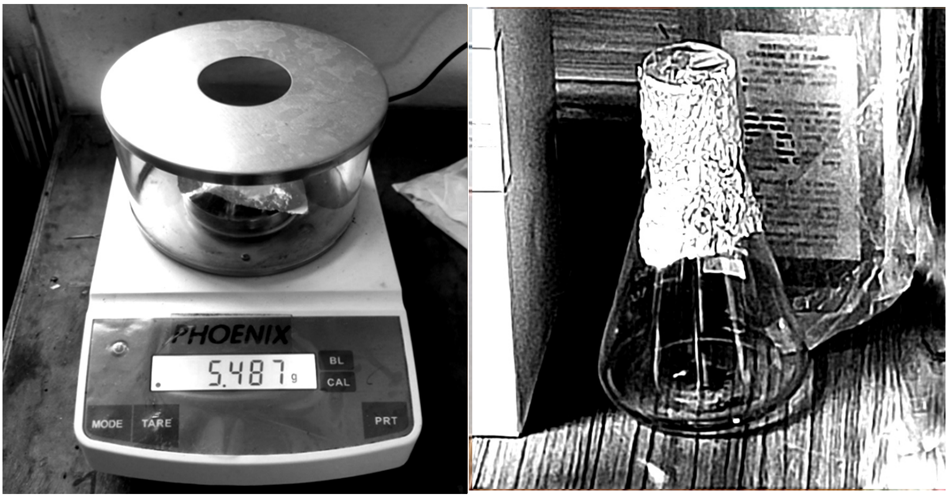

Fig. 1. (a) Electronic weighing machine (b) Conical flask consisting of Zinc acetate dihydrate solution

The weighted Zinc Acetate was mixed with 50 ml isopropyl alcohol (C_3H_8O). The solution was stirred until all the salt was dissolved in a 50 ml solution of isopropyl alcohol. The solution was kept in the temperature-maintained furnace. The solution is then stirred at 60^0 centigrade for two hours. After the heating process, the solution was taken out and allowed to cool down slowly. 5 to 6 drops of di-ethanol amine ($C_4H_{11}NO_2$) were added dropwise, and a curdy white precipitate was filtered using filter paper. The obtained clear transparent solution was sealed with Aluminium foil and was kept in a dark place for 24 hours.

Preparation of Aluminum acetate nanohydrate solution

The work is associated with the study of the Al-doped ZnO thin film, so for Aluminium doping another solution was prepared and for the preparation of a solution of Aluminium nitrate nonahydrate (Al $(NO_3)_3 \cdot 9H_2O$)). was dissolved in 50 ml isopropyl alcohol. The prepared solution was heated in a furnace at 60 degrees for two hours. 5 to 6 drops of Di-ethanol amine were added to the heated solution. The formed precipitate was filtered using filter paper clear transparent solution was kept in a dark place for 24 hours.

The prepared 50 ml Zinc acetate solution was taken in 5 test tubes each 10 ml. For 1 percent concentration, 100 microliters of Aluminium acetate nano hydrate solution were added to 10 ml zinc acetate solution. Similarly, for (2, 3, 4, 5) percent concentration, (200, 300, 400, 500) microliter zinc acetate solution was added to each 10 ml solution. Each 1,2, 3, 4, and 5 concentration solution was coated with the help of a spin coater and heated with the help of a furnace. The process was repeated for 10times**.**

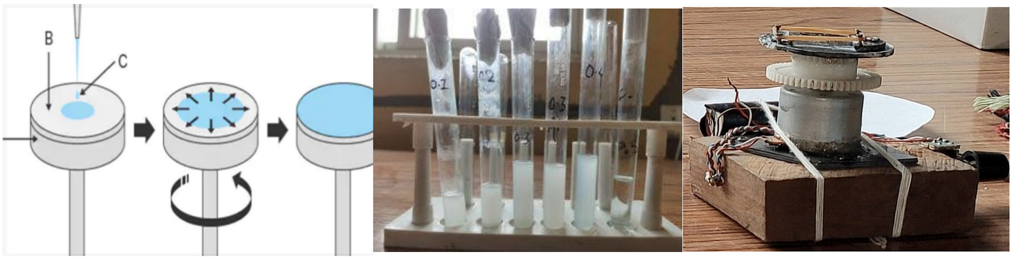

Fig. 2. (a) Spin coating technique (Akhtar *et al.*,2015), (b) Aluminium added ZnO solution b) Spin coater.

Spin coating technique

The sol-gel process or the spin coating technique is one of the attractive techniques used for film deposition. It has low equipment cost, no vacuum requirement, low-temperature processing, and is easy to dope and use. The thin films by sol-gel technique have good homogeneity and excellent compositional control with good electrical and optical properties. It is well known that the film properties deposed by the sol-gel technique are determined by preparation parameters area coating (Gul, 2012).

Figure 2(a) shows the schematic diagram of the spin coater and how it works. Whereas Figure 2(c) shows the homemade or self-made spin coater. The spin coater in the figure is provided with two batteries of 3.7 V each connected in a series connection. The device consists of a platform to place the sample and two clips to hold the sample from falling while spinning. Also, a variable potentiometer is provided to change the rotating speed of the motor.

Fabrication of undoped ZnO thin film

First, the glass substrate was kept on the rotator (spin coater) and fixed within the platform. After that, a dropwise solution of Zinc acetate dihydrate was dropped on the substrate and the rotator was spun at 1800 rev/min for 40 seconds. Then the substrate was taken out and heated up to the temperature of 300^0 Centigrade and cooled down slowly.

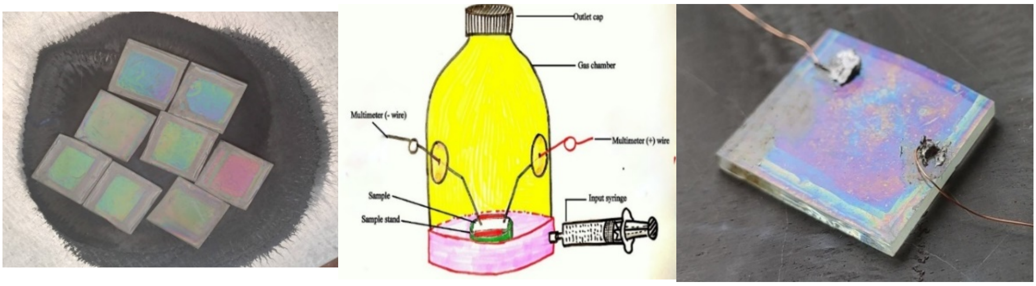

Fig. 3. (a) Al-doped ZnO thin film (1% concentration), (b) Gas sensing mechanism c) ZnO thin film connected with wires.

The deposited glass substrates are again doped with the prepared solution and the process of spin coating, heating, and cooling is done about 10 times. Thus, the fabrication of doped and undoped ZnO thin film was done.

Gas Sensing Mechanism and Characterization

For the gas sensing mechanism, a homemade gas sensor was used which consists of an input hole from which liquid acetone was passed with the help of a 5 ml syringe. Also, it consists of an outlet hole from which extra gas is released. There are two connectors to touch the sample for measuring DC resistance. The sample was kept on the sample holder which was fixed at the bottom of the gas chamber. The two wires coming from the chamber were connected to a digital multimeter which was used to measure resistance. The gas sensing mechanism is primarily based on electron transfer between absorbed oxygen species and the molecules of the test gas. The sensor's response is largely influenced by the quantity of absorbed oxygen species, as well as the specific surface area, structure, active sites, and electron characteristics of the sensing material. The detection of gas is accomplished by observing changes in the sensor's resistance. atmospheric oxygen on the oxide surface. This process extracts electrons from the semiconductor, resulting in changes in carrier density and conductivity. The interaction oxidizing or reducing gases alters the oxygen concentration and conductivity, enabling the measurement of gas concentration. The effects of oxidizing and reducing gases are opposite to each other.

UV visible spectrophotometer

The device shown in the figure below was used for the determination of absorbance and transmittance.

Fig. 4. (a) UV-V spectrophotometer, (b) Digital multimeter.

First, the prepared sample was kept inside the spectrophotometer and the wheel was rotated to change the wavelength (nm) and respective absorbance and transmittance were recorded. UV-V is spectrophotometry, an electronic device used in various research fields. It is usually in the laboratory, and it is a laboratory technique used in the measurement of absorbance of light across ultraviolet and visible regions. Its working mechanism is that when the incident light strikes the sample, it is usually reflected, transmitted, or absorbed. The absorbance of light causes the transition of molecules from the ground state to the excited state. UV-V is a spectrophotometer, an analytical instrument, that serves this principle and measures the intensity of light absorbed by the molecule. The detector detects the reading, and the values are shown in the display attached to it.

Results and Discussion

For the study of the optical properties of doped and undoped ZnO thin film, a spectrophotometer was used for measuring different parameters such as absorbance, and transmittance. For the calculation of bandgap and refractive index, the graphical software called Origin Pro 2021 was used. The UV- spectrophotometer was used to study the optical properties of doped and undoped ZnO thin film at various Aluminium doping concentrations (1%, 2%, 3%, 4%, and 5%) to study the transmittance T% and absorbance (au) spectra which are used to investigate optical properties that lead to understanding the optoelectronic nature for these films. The device spectrophotometer was used for measuring the absorbance of the ZnO thin film. The absorbance for undoped and doped ZnO thin film was measured, where data for different concentrations of Al-doped ZnO were taken and a graph was plotted with the help of OriginPro 2018 SR1 v9.5.1.195 x86-x64.

Optical Properties of Al dopped ZnO

The wavelength and absorbance spectra of undoped and Al-doped ZnO thin films with different concentrations are presented in Figure 5 (a). The absorbance at different wavelengths was measured at room temperature. The result indicates the absorbance spectra can be interpreted in terms of mainly two regions. The region that has high photon energies contains the absorption edges of the undoped and Al-doped ZnO thin film and is characterized by its relatively higher absorbance as shown in Figure 5 (a). The second region lies in the visible region and the spectra are characterized by the low absorbance. In addition, Figure 5 (a) indicates that the edge of absorbance is shifted upward as the Al-concentration is increased.

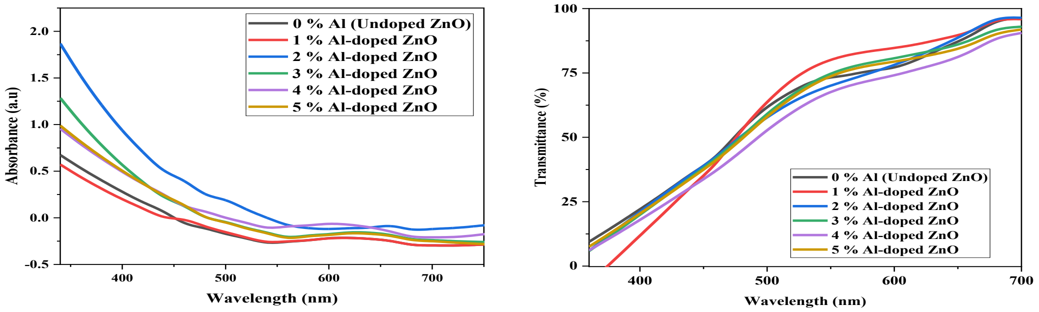

Fig. 5. (a) Absorbance of undoped, and 1%, 2%, 3%, 4%, 5% Al-doped ZnO, and (b) Transmittance undoped, and 1%, 2%, 3%, 4%, 5% Al-doped ZnO.

The above graph depicts the variation of optical absorbance and the wavelength. Where the wavelength is measured along the x-axis and absorbance is measured along the y-axis. The absorbance is higher at the wavelength ranges from 350 nm to 400 nm, and there is a gradual decrease in absorbance as its wavelength increases gradually. It was found that the absorbance is higher in the UV region and lower in the visible region. Also, there is an inverse relation between wavelength and absorbance was the same for different concentrations of Al-doped ZnO thin film.

It is observed that the 2% and 3% Al-doped ZnO thin film showed higher absorbance whereas undoped, 1%, 4%, and 5% Al-doped ZnO thin film showed lower absorbance this may be because thin film effects due to interference occur that can not only lead to unexpected absorbance values (Mayerhofer, 2017).

The optical transmittance (T%) spectra of undoped and doped ZnO thin films with different Aluminium concentrations are presented in Figure 5 (b). The optical spectra are measured at room temperature. The result indicates that the transmittance spectra can be interpreted in terms of mainly two regions. The first has high photon energy i.e., UV region, and is characterized by its relatively lower transmittance as shown in Figure 5 (b). The second region lies in the visible region and these spectra are characterized by its higher transmittance and no light absorbance. Also, it showed that as the Al-concentration is increased then the transmittance spectra are shifted upward.

Figure 5 (b) depicts the variation of optical transmittance (T%) of ZnO thin film in the wavelength region 400 nm to 700 nm. The average transmittance of the film, including substrate in the visible range is observed over 75%. The value of transmittance suddenly drops close to zero when the wavelength falls below 300 nm because of band gap absorption. The transmittance is higher in 1%, and 2% Al-doped ZnO thin film whereas 3%, 4%, and 5% Al-doped ZnO thin film show less transmittance which may be due to a similar case as in the absorbance (Mayerhofer, 2017).

Band gap of undoped and Al-doped ZnO thin film

The value of energy was calculated with the help of the formula as shown in the equation below.

$$E = \frac{1240}{\lambda} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(iii)}$$

Where E is energy and λ *is* wavelength

The absorption coefficient was calculated using the formula given below.

$$\alpha = (E \times Absorption)^2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (iv)$$

Where $\boldsymbol{\alpha}$ (Alpha) is the absorption coefficient and E is the energy

The band gap (E_g) of ZnO films was calculated from the tauc-plot described by the equation:

$$\alpha \mathrm{h}\nu\, 2 = A(\mathrm{h}\nu - E) \ldots\ldots\ldots\ldots\ldots\ldots (v)$$

where α, A, hν, and Eg are the absorption coefficient, energy constant, photon energy, and band gap respectively (Cheng & Ma, 2009).

The calculated value of absorption coefficient and energy calculated with equation (i) for different concentrations are plotted with the help of OriginPro 2018 SR1 v9.5.1.195 x86-x64 and the graph is shown in figure 6(b).

The results showed E_g increases from 3.208 eV for undoped to 3.385 eV for 1% Al-doping sample. This increase in E_g due to Al doping was found to be consistent with the reports on similar systems due to a decrease in the grain size of MOS film [28]. If Al^{3+} ions substitute the Zn^{2+} ions from their lattice sites, the additional free charge carriers were added which were responsible for shifting the Fermi level into the conduction band to increase the band gap of Al-ZnO films.

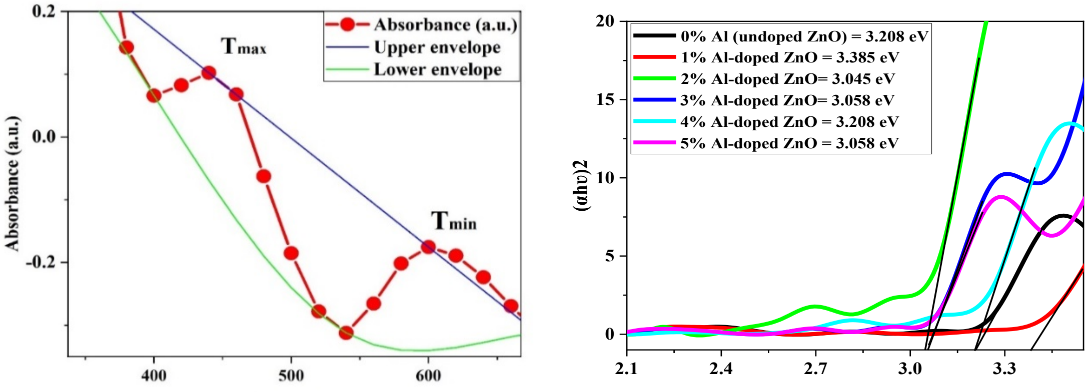

Fig. 6. (a) Upper and lower envelope of absorption spectra (tauc-plot) [41] (b) Absorption coefficients of undoped, and 1%, 2%, 3%, 4%, 5% Al-doped ZnO

Figure 6(b) depicts the variation of absorption coefficient and energy. The graph between energy and absorption coefficient (Alpha) is plotted which is used to calculate the band gap of different ZnO thin films of different concentrations. It found that the band gap of the undoped ZnO thin film was found to be 3.208 eV, and for Al-doped ZnO thin film of 1 percent concentration was found to be 3.385 eV, for 2 percent concentration it was 3.045 eV and for 3% concentration was 3.058 eV, for 4% concentration it was found to be 3.208 eV and similarly for 5% concentration was found to be 3.058 eV.

Refractive index of Al-doped ZnO (Different concentration)

Measuring the refractive index 'n' is essential for the optical characterization of optical devices such as optical switches, filters, modulation, and other optoelectronic devices. The refractive index n of thin films can be calculated from the formula, $n = \sqrt{N + \sqrt{N^2 + n_s^2}}$. The resultant n as a function of incident light wavelength is shown in Figure 7 (a) and (b).

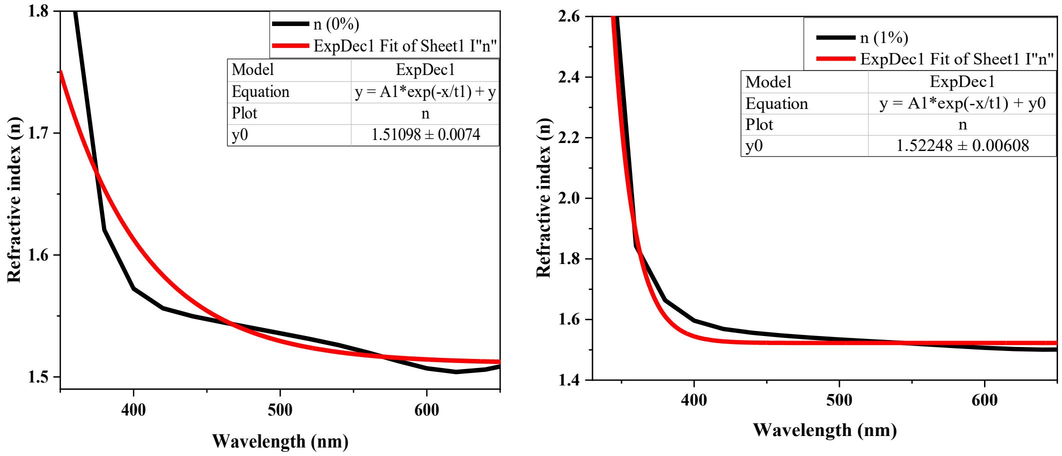

Fig. 7. (a) Refractive index spectra of undoped ZnO as a function of wavelength (b) Refractive index (1% Al).

Our results indicate that the refractive index has a normal dispersion in high-wavelength regions followed by an anomalous dispersion in the low-wavelength zones. Moreover, Figure 7(a), demonstrates that in a strong absorption region (λ < 560 nm), the refractive index exhibits higher values. In the region, λ ≥ 560 nm, the refractive index decreases sharply with wavelength and shows significant normal dispersion. We found that the refractive index of the undoped ZnO exhibits values ranging between 1.479 to 1.522 and as wavelength decreases from 700 to 350. However, as the aluminium concentration increases, the refractive index increases. Similarly, for different Al-doped ZnO thin films, the refractive index varies. So, for 1% Al-doped ZnO thin film, the refractive index was 1.522. Similarly, for 2% Al doped ZnO thin film, the refractive index was found to be 1.492.

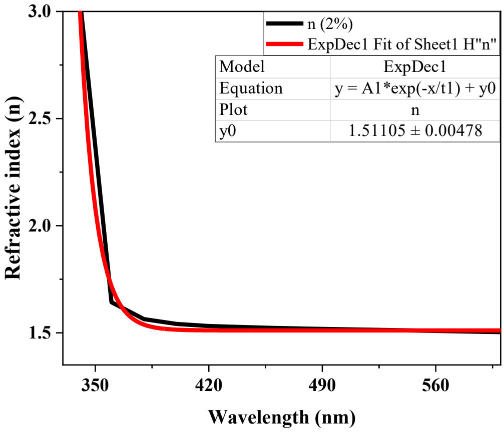

Fig. 8. (a) Refractive index (2% Al).

The decrease in the refractive index of the film may be due to impurities present in the thin film and the increase in the refractive index of Al-doped ZnO thin films can be explained in terms of the formation of clusters of atoms as the Aluminium is injected into ZnO matrix during the sol-gel formation.

Conclusion

This study provides a comprehensive analysis of aluminum-doped zinc oxide (ZnO) thin films, focusing on their optical, electrical, and gas-sensing properties. The 1% and 2% Al- ZnO sample showed a better response than the undoped ZnO film. The increase in the percentage of dopant i.e., Aluminium in the ZnO thin film, the sensitivity was somewhat increased. Time-dependent drift in resistance of ZnO films has been observed. ZnO films showed a fast and slow resistance change response when exposed to gases with varying volumes. The maximum values of sensitivity in terms of resistance were found to be 199.20 for doped and 189 for Al-doped and undoped ZnO thin film for 3 ml volume. The decreased resistivity with increasing Al concentration may be attributed to an increase in both carrier concentration and Hall mobility. The optical analysis revealed that higher aluminum doping levels enhance the transmittance of ZnO films, with 1% Al-doped ZnO exhibiting the highest transmittance of approximately 70%. The refractive index and extinction coefficient analysis further confirmed that higher doping levels reduce light scattering and absorption, resulting in superior optical properties. The electrical characterization indicated that aluminum doping decreases the conductivity of ZnO thin films. Notably, 2% Al-doped ZnO films exhibited consistently higher sensitivity, suggesting an optimal doping concentration for gas sensing applications. The long-term stability of ZnO nanostructures-based gas sensors is another challenge attributed to changing environmental conditions like different temperatures and different humidity levels. Such types of effects should be minimized using keeping the same environmental condition near the gas sensors in real-time monitoring. The fabrication of highly selective gas sensors based on ZnO nanostructures is one of the most important tasks.

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