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Physical Properties of Zno: Ga_2O_3 Thin Film Prepared by Microwave-Assisted Pyrolysis: The Role Ga_2O_3

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ABSTRACT

We have successfully developed high-quality polycrystalline ZnO: Ga_2O_3 thin films using the microwave-assisted spray pyrolysis method. The variation of Ga_2O_3 doping (0-6%) resulted in significant changes in the morphology, crystal structure, and optical properties of the thin films. The 3% Ga_2O_3-doped sample exhibited excellent crystal quality with the smallest Full Width at Half Maximum (FWHM) value of 0.16°, high transmittance (96%), and a suitable band gap (3.21 eV) for applications in sensor systems, optoelectronic devices, and solar cells. This study presents a significant advancement in the development of high-quality ZnO: Ga_2O_3 thin films with superior characteristics for various applications.

Keywords: Thin film; ZnO: Ga_2O_3; microwave asisted spray pyrolisis

INTRODUCTION

In recent decades, the development of functional materials has grown rapidly and opened up many opportunities for innovation in various fields of technology. One such material is thin film material with various unique characteristics and has various advantages when compared to conventional materials [1]. Thin films continue to attract considerable attention from researchers due to their great potential, especially in the fields of sensors, solar cells, semiconductors, and other applications[2][3][4][5][6]. These materials are used in electronic nanotechnology due to their unique properties, such as high conductivity, stability, and flexibility, making them suitable for various applications [7].

Thin films are layers of material ranging in thickness from nanometers to micrometers that coat the surface of a substrate [8]. In the manufacturing process, the selection of the type of material is one of the most important parts to obtain more specific properties and functions, so as to improve its performance and effectiveness in various applications. One promising material in thin film manufacturing is zinc oxide (ZnO), which is an n-type semiconductor with a wide band gap (3.37 eV) and high thermal and radiation stability [9][10]. ZnO is widely used in optoelectronic devices, gas sensors, and solar cells because it is affordable, non-toxic, and environmentally friendly [11][12]. However, pure ZnO has limitations such as low electrical conductivity and limited corrosion resistance, which requires modification by doping [13][14].

To improve the electrical conductivity of pure ZnO, thin films are often doped with group-III elements like Aluminum (Al) [15], Indium (In) [16] and Gallium (Ga) [17] or group-II elements such as Calcium (Ca) [18] and Magnesium (Mg) [19]. In particular, doping ZnO with Gallium Oxide (Ga_2O_3) has been shown to be effective in improving electrical properties, reducing resistivity, and increasing chemical stability [20]. Ga_2O_3 has a band gap energy of 4.9 eV and is able to induce minimal lattice distortion because its atomic size is similar to ZnO [21]. In addition, Ga_2O_3 can also improve the crystallinity and morphology of thin films, making it suitable for optoelectronic and piezoelectric applications [22][23]. Previous studies have shown that ZnO: Ga shows high transmittance (up to 89%) and excellent structural stability [24]. Therefore, Ga_2O_3 doping is a promising solution to overcome the limitations of pure ZnO.

Various deposition methods have been developed to produce high-quality thin films, including sputtering [10], sol-gel [12], spray pyrolysis [25], pulsed laser deposition [26], and aerosol assisted chemical vapour deposition

[27]. Most of these methods require high vacuum, high temperature, expensive equipment, catalysts, and the use of toxic gas compounds [28]. Among these methods, microwave-assisted spray pyrolysis is an attractive option because by using simple equipment, economical, and easy operation, it can produce uniform heat distribution, accelerate precursor decomposition, and produce films with uniform morphology [29][30]. The microwave assisted spray pyrolysis method requires low cost and environmentally friendly processes so that its use is more efficient than conventional methods [31]. In this method, the interaction of microwaves with the substrate allows optimal film growth at lower temperatures [32].

The aim of this study is to investigate the growth of ZnO:Ga_2O_3 thin films with varying Ga_2O_3 doping concentrations using the microwave assisted spray pyrolysis method. The main focus is to elucidate the effect of doping on the morphology and crystal structure of the films. The microwave assisted spray pyrolysis method has been chosen because of its simplicity and high efficiency in producing high quality thin films with superior electrical and optical properties, thereby making a significant contribution to the development of future semiconductor materials.

MATERIAL AND METHODS

Thin films were grown on a silica (Si) substrate by microwave assisted spray pyrolysis. Ga_2O_3 was doped into the ZnO solution via zinc acetate dihydrate as the main precursor, gallium (III) oxide as the dopant, isopropanol as the solvent and ethanolamine as the stabilizer. Solutions were prepared with a mass percentage of Ga_2O_3 of 0%, 1%, 2%, 3%, 4%, 5% and 6% of the mass of ZnO. Precursor and dopant solutions were prepared at a concentration of 0.5 M in 20 mL of isopropanol and stirred at 70°C for 60 minutes using a magnetic stirrer. The final solution was white, very homogeneous and transparent [33]. The clean and dry substrate was placed in a microwave oven set at medium high plate temperature for 10 minutes to preheat. The ZnO:Ga_2O_3 solution was then coated onto the substrate using the spray pyrolysis method. The substrate coated with the ZnO:Ga_2O_3 solution was left in the microwave for 20 minutes and then annealed. The annealing process was carried out in a furnace for 1 hour with a holding temperature of 450°C [32]. The resulting thin films were then characterised using scanning electron microscopy (SEM), energy dispersive X-ray (EDX) and UV-Vis spectrophotometer.

RESULTS

The ZnO:Ga_2O_3 thin film deposition in this study uses a pressure-based spray gun as an atomiser. Atomisation is the process of breaking the precursor solution into small droplets resembling mist due to the effect of pressure as the liquid exits towards the substrate [34]. This process is assisted by nitrogen gas pressurised at 20kg/cm² (2atm) to form fine droplets. The droplets are heated by interaction with electromagnetic waves from the microwave, resulting in faster methanol evaporation compared to conventional spray pyrolysis methods [35]. This faster evaporation is due to the polar nature of methanol, which is sensitive to dipole reactions caused by microwave radiation [36].

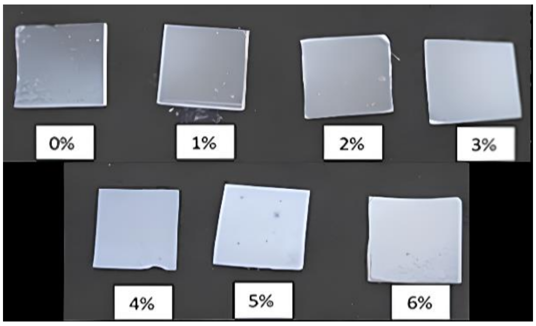

Figure 1. Deposition results of pure ZnO and ZnO:Ga_2O_3 thin films.

The deposition results of ZnO:Ga_2O_3 thin films with different masses of Ga_2O_3 dopants are shown in Figure 1. ZnO films without dopants have the highest transparency, while increasing the mass of dopants causes the film to become less transparent. This decrease in transparency is due to the increase in the number of constituent atoms in the film, which increases the number of collisions of light particles with these atoms, making it more

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difficult for light to pass through the film [37]. The thickness of the film affects optical properties such as absorption, transmission and attenuation. The thicker the film, or the higher the dopant concentration, the higher the absorbance and attenuation constants, while the transmittance decreases [38][39].

Morphology

Scanning electron microscopy (SEM) characterisation was carried out to study the surface morphology of $ZnO:Ga_2O_3$ thin films. SEM works by scanning a sample with a high energy electron beam, which produces a signal containing information about the surface topography [40]. The SEM results in Figure 2 show that the addition of Ga_2O_3 doping affects the surface morphology of ZnO thin films. In the pure ZnO thin film, cracks are found which are caused by the very thin film thickness. The addition of Ga_2O_3 doping concentration from 1% to 6% causes the film surface to become more homogeneous with fewer cracks. The increased thickness also contributes to the improvement of the morphological structure of the thin film [41]. These morphological changes have a significant impact on the optical and electrical properties of the film, which are highly relevant for optoelectronic applications.

Based on the SEM characterisation results in Figure 2, it can be observed that increasing the Ga_2O_3 doping not only reduces the number of cracks on the surface of the ZnO thin films, but also improves the homogeneity of the structure. At higher doping concentrations, more uniform grain growth is observed, indicating an improvement in the crystallinity of the material. This denser structure and lack of cracks may contribute to increased electron mobility, thereby improving the electrical conductivity of $ZnO:Ga_2O_3$ thin films [42].

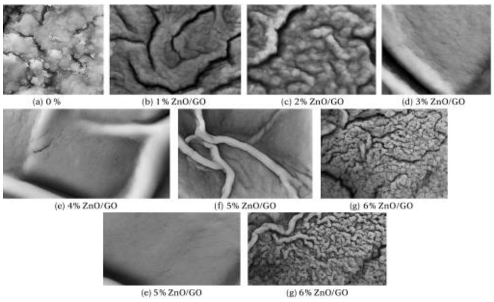

Figure 2. Sem results with 25000x magnification

Energy dispersive X-ray (EDX) characterisation was used to analyse the chemical composition of $ZnO:Ga_2O_3$ thin films. The principle of EDX is to detect X-ray fluorescence signals emitted when high energy electrons strike the sample [42]. EDX analysis reveals the presence of key elements such as Zn, O and Ga, which come from the precursor material. Other elements such as Si come from the glass substrate used to prepare the sample, while N comes from the nitrogen gas used as an inert gas during the deposition process. The elements Ca and Mg are likely to come from environmental contamination during the deposition process.

EDX characterisation shows an increase in the content of Ga atoms with the addition of dopants, which is not detected in pure ZnO films (without dopants). The presence of Ga in the ZnO film contributes significantly to improving the chemical stability and reducing the oxidising properties of pure ZnO. In addition, Ga as a dopant effectively improves the morphological quality and crystal structure of the film, which affects its optical and electrical performance [43]. Thus, the combination of SEM and EDX characterisation shows the successful deposition of high quality $ZnO:Ga_2O_3$ thin films.

Table 1. Chemical elements that form the $ZnO:Ga_2O_3$ thin film

Ga_2O_3 (%)	Chemical Element (Atomic)						
	Zn	O	Si	Ga	N	Ca	Mg
0	19.12	47.93	19.18	-	11.63	1.89	0.26

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1	39.29	43.17	5.59	0.17	11.07	0.71	-
2	42.79	43.19	3.24	0.43	10.25	0.11	-
3	40.92	44.37	3.74	0.37	10.18	0.42	-
4	36.03	22.77	2.95	0.31	11.75	0.35	-
5	86.91	10.49	-	0.45	2.08	0.06	-
6	42.88	47.10	0.10	0.57	9.35	-	-

Crystal Structure

X-ray diffraction (XRD) characterisation was carried out to qualitatively and quantitatively identify compounds in ZnO:Ga_2O_3 thin films. The XRD data were obtained in the form of diffractograms, which are plots of the relationship between intensity (I) and diffraction angle (2θ), and provide information on the crystal structure, planar orientation, full width at half maximum (FWHM), grain size and Bragg plane spacing (d-spacing) [9]. The XRD results in Figure 3 show that the ZnO:Ga_2O_3 thin film has a polycrystalline structure with different diffraction patterns for each doping variation. The diffraction pattern shows peaks in the (100), (002) and (101) plane orientations, which are characteristic of the hexagonal structure of ZnO (ICDD 01-078-3315). The addition of Ga_2O_3 doping produced new diffraction peaks indicating the presence of a Ga_2O_3 phase with an orthorhombic structure. In addition, the diffraction peak in the (002) plane of the glass substrate (Si) decreases in intensity as the Ga_2O_3 doping concentration increases. This is due to the distortion in the crystal lattice due to the substitution of Zn^{2+} ions with Ga^{3+} ions [44].

At 3% Ga_2O_3 doping, the diffraction in the (002) plane experiences the highest intensity, indicating a preferential orientation perpendicular to the substrate. The increase in intensity in this plane is comparable to the results of the study showing that the (002) orientation supports the application of thin films for photoanodes in dye sensitised solar cells (DSSC) and sensor systems. The addition of doping up to 6% also affects the crystal quality, with a reduction in cracks and an increase in surface homogeneity.

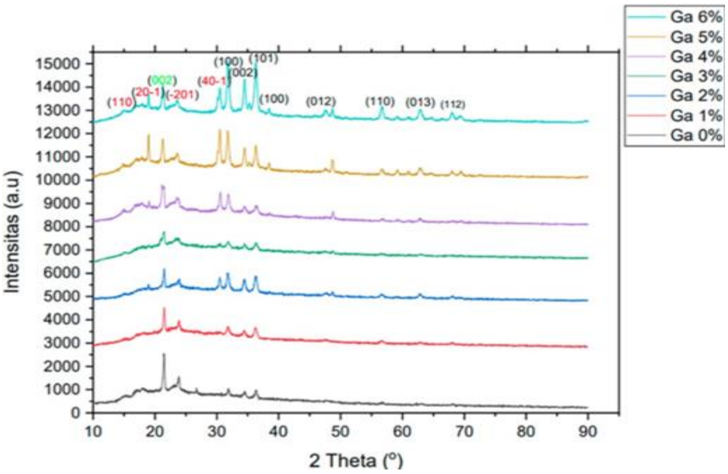

Figure 3. XRD spectrum ZnO: Ga_2O_3 Thin film

Table 2 shows the FWHM, d-spacing and grain size for each Ga_2O_3 doping variation. The diffraction angle (2θ) is in the range of 31°, with a shift towards smaller angles up to 2% doping, then increasing at 3% doping before decreasing again. This shift shows the influence of lattice strain on the crystal structure. The D-spacing is inversely proportional to the diffraction angle, with the smallest value at 3% doping, indicating the best crystal quality. The ZnO:Ga_2O_3 thin film with 3% doping has the smallest FWHM (0.16°) and the largest grain size (51.64 nm), indicating that this doping produces the best quality crystals. Good crystals have a narrow FWHM and a large grain size [45]. In contrast, the addition of 5% and 6% doping causes the grain size to decrease due to the increased film thickness and less homogeneous surface, which affects the crystal quality. XRD analysis

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results show that the addition of Ga_2O_3 doping successfully improves the crystalline quality of ZnO thin films at the optimum doping concentration of 3%. The significant presence of Ga_2O_3 phase in this thin film contributes to the improvement of optical and electrical properties, making it suitable for sensor applications and optoelectronic devices.

Table 2. Crystal analysis

No	Ga_2O_3 (%)	2θ (°)	FWHM (°)	*d-spacing* (Å)	*Grain size* (nm)
1	0%	31.87	0.3287	2.8059	25.1346
2	1%	31.84	0.4972	2.8087	16.6153
3	2%	31.76	0.5524	2.8171	14.9520
4	3%	31.88	0.1600	2.8076	51.6373
5	4%	31.80	0.1700	2.8121	51.6270
6	5%	31.79	0.5444	2.8147	15.1729
7	6%	31.79	0.5351	2.8147	15.4366

Optical Properties

ZnO:Ga_2O_3 thin films are materials with thicknesses ranging from angstroms to microns. The optical properties of this material are strongly influenced by its composition and thickness. Characterisation was carried out using UV-Vis spectroscopy at wavelengths of 200-1200 nm to measure absorbance, transmittance and to determine the band gap. The test results in Figure 4 show that ZnO:Ga_2O_3 thin films have high absorbance in the 200-400 nm range, which then decreases at longer wavelengths. The addition of Ga_2O_3 doping increases the absorbance up to 2% doping concentration, but decreases at 3-5% concentration and increases again at 6% doping. This variation is related to the thickness of the thin film according to the Lambert-Beer law, where the absorbance is directly proportional to the thickness of the material [46]. The shift of the absorption wavelength towards higher wavelengths with increasing Ga_2O_3 concentration indicates a modification of the optical spectrum of the material, in accordance with the study.

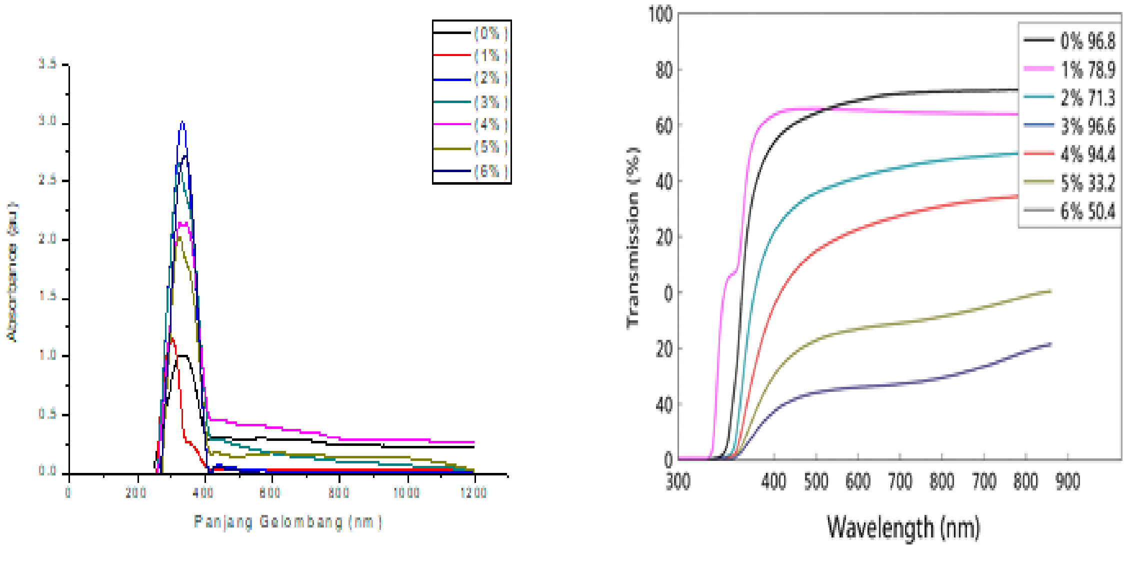

Figure 4. Plot of absorbance & transmittance of ZnO:Ga_2O_2 vs. Wavelength

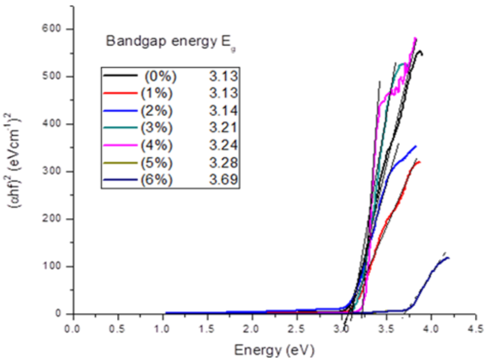

Figure 5. Graph of gap band ZnO:Ga_2O_2 thin film

The transmittance of the material shows a decreasing trend with increasing Ga_2O_3 doping, from 98.8% in pure ZnO to 71.3% at 2% doping. However, the transmittance increases significantly at 3-4% doping (96.6% and 94.4%) before dropping dramatically at 5-6% doping (33.2% and 50.4%). This decrease is due to the increase in thickness and surface inhomogeneity which affects the optical properties of the thin film [47]. Band gap measurements using the Tauc plot method in Figure 5 show that the addition of Ga_2O_3 broadens the band gap, known as the Moss-Burstein effect. The pure ZnO film has a band gap of 3.13 eV, while doping with 6% Ga_2O_3 increases the band gap value to 3.69 eV. Annealing at 450°C for one hour also improved the crystal quality, resulting in quantum effects on the nanocrystalline structure and band gap widening [32]. The increase in band gap and the control of transmission make ZnO:Ga_2O_3 thin films potentially applicable to sensor systems, optoelectronic devices, LEDs and solar panels.

Potential of thin film ZnO: Ga2O3

ZnO thin films have attracted attention due to advantages such as low price, environmental friendliness and easy deposition process. However, pure ZnO has disadvantages such as high resistivity ($1.18 \times 10^{-10} \Omega$ cm) and low conductivity ($8.47 \times 10^{-11} (\Omega$ cm$)^{-1}$), and is less stable in corrosive environments [48][49]. In this study, the addition of Ga_2O_3 doping is used to overcome these shortcomings.

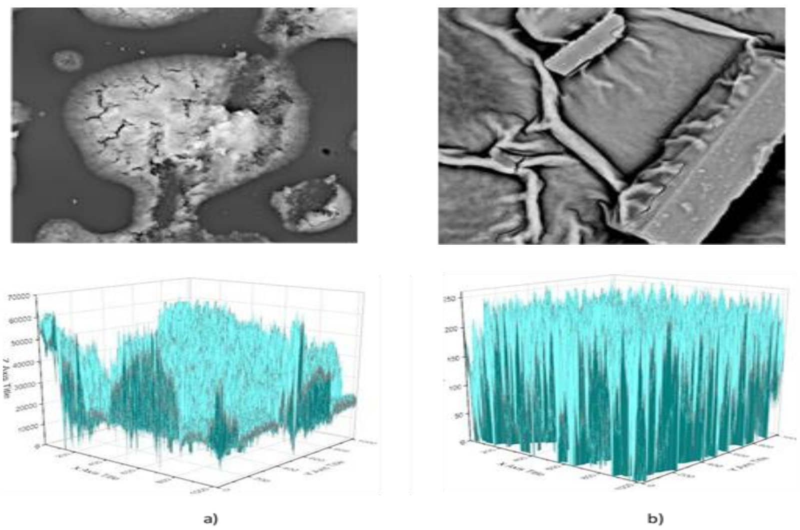

Figure 6. Result of thin film SEM and 3D modelling with origin a) pure ZnO; b) ZnO:Ga_2O_2

The results show that Ga doping improves the adhesion of the film to the substrate and increases the homogeneity of the surface morphology, as shown in Figure 6. Pure ZnO has many cracks and pores, while Ga_2O_3 doping results in a more compact and uniformly distributed morphology. Figure 7 shows the porosity modelling of ZnO and ZnO thin films: Ga_2O_3 where the porosity of pure ZnO thin films reaches 57%, while Ga doping reduces the porosity by filling the pores, increasing the density and mechanical strength. However, excessive doping (≥6%) makes the film too thick and reduces transparency.

The optimum doping was found to be 3%, with the smallest FWHM value (0.16), indicating a larger crystal size and lower lattice strain [50]. These results indicate that modification by Ga_2O_3 doping improves the optical, mechanical and adhesion properties of ZnO thin films, making them more suitable for optoelectronic applications such as solar cells and sensors.

CONCLUSION

The results showed that ZnO:Ga_2O_3 thin films were successfully fabricated on a substrate using the microwave assisted spray pyrolysis method with controlled parameters. SEM-EDX characterization shows that the addition of Ga_2O_3 doping can increase the compactness and thickness of the surface structure, while XRD characterization shows a polycrystalline structure with orientation on several crystal planes. Spectrophotometric UV-Vis analysis shows that the thin films have an absorbance in the 200-400 nm wavelength range, a transmittance of up to 98.8% and a band gap varying from 3.13 eV to 3.69 eV. In particular, the film with 3% Ga_2O_3 doping shows optimal performance with the smallest FWHM (0.16˚), largest grain size (51.6373 nm), high transmittance (96%) and ideal band gap of 3.21 eV, making it very promising for sensor applications as well as optoelectronic devices.

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