Scientific Research Journal,
Vol 18, No 2 (2021) 147-160
https://doi.org/10.24191/srj.v18i2.13035

Physical Properties of Different Thicknesses ZnO Thin Films Prepared via Sol-gel Spin Coating Technique

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Received: 5 April 2021
Accepted: 15 July 2021
Online First: 31 August 2021

ABSTRACT

This paper presents the investigation of the thickness of the ZnO thin films by varying the number of deposition layers during the spin coating deposition process. ZnO thin films were deposited with a different number of layers (ranging from 1, 3, and 5), and the main purpose of this study is to explore the effect of the thickness on the properties of ZnO thin films. The deposited thin films were characterised using field emission scanning electron microscope, surface profilometer, and X-ray diffraction. From the characterisation results, the morphology of the ZnO thin films changed significantly with the number of layers and their thickness value. As expected, the thickness increased as the number of layers increased. The crystalline quality of the deposited film improved as the thickness increased. A change in crystallographic orientation was also observed in which the thicker, thin films showed crystal growth in the (102) direction, whereas the thinner one was in the (101) direction. A slight increase in crystallite size for dominant orientation also was observed with the increase of film thickness.

Keywords: sol-gel, spin coating, nanostructures, annealing

INTRODUCTION

In recent years, semiconductor thin films have received intense research interest among researchers due to their wide potential in various applications [1-3]. In between all available semiconductors, zinc oxide (ZnO) has shown great potential as a promising candidate for application in many applications, including electronics and optoelectronics devices. ZnO is a type of semiconductor material that has a direct band gap of 3.37 eV with hexagonal wurtzite structure and belongs to group II-VI having a large exciton binding energy (60 meV) [4]. In addition, ZnO is also one of the richest families of nanostructures, demonstrating many types of nanostructures such as nanorods, nanobelts, nanoflowers, tetrapods, and nanofiber [5-7]. All of these nanostructures have novel applications as sensors, biomedical sciences field, as well as transducers.

More interestingly, ZnO can be prepared using various methods and techniques. For instance, physical vapor deposition (PVD) [8], pulsed laser deposition (PLD) [9], dip coating [10], and chemical bath deposition (CBD) [11]. Among all, the sol-gel spin coating technique is favorable among researchers due to its versatility. Sol-gel technique offers a low-cost method, is environmentally friendly, and can be conducted at relatively low temperatures [12]. The sol-gel method is based on the wet chemistry process that involves the preparation of sol, the gelation of a sol, and the removal of the liquid existing in fine interconnected channels within the gel [13]. While the spin coating technique is a common method used to coat the prepared solution onto the desired substrate, the primary advantage of this technique is it can quickly and easily produce a very uniform thin film [14].

By using this technique also, the thickness of the thin film could be easily adjusted. The concentration of the prepared sol-gel, spinning condition, duration, and heat treatment duration, and temperature are some of the factors that will determine the thin film thickness. Other than these factors, increasing or varying the number of layers during the deposition process also will alter the thickness of the thin film produced. The thickness of the thin film plays an important role depending on the application chosen [15]. For example, electrical properties will be improved when the thin film has a higher thickness value due to the improvement in crystallinity. A suitable thickness will decrease the resistivity, thus improve the electrical

properties [16]. However, if the film has a very high thickness, it also will affect the electrical properties due to the increase in resistivity. Other than that, the thickness of the thin film also would influence the structures and the morphology of the films. E. Mohajerani et al. reported that their polymer blends structures constantly change when the film thickness increases [17].

In a reported study conducted by N. Mufti *et al*., ZnO thin films were spin-coated at different spin speeds (2000-4000 rpm), produced different thickness values, in the range of 0.2 – 1.0 μm [18]. Besides thickness, the crystal size of spin-coated ZnO thin films is also affected by the thickness of the thin film. U. Chaitra *et al*. also explored the effect of thickness on their ZnO properties [19]. Sol-gel spin coating technique had been chosen to deposit their ZnO thin films; thus, the molarity of the precursor had been varied. Different molarity had produced different thicknesses, and the optical and electrical of the thin films were affected by this thickness. Based on these studies, it could be concluded that the thickness of the deposited thin films would affect and influence the properties of thin films. To prove this statement, in this study, the effect of the film thickness was investigated by varying the number of layers during the spin coating deposition process. The different layers of ZnO thin films were characterised to study how the thickness would affect their physical and structural properties.

METHODOLOGY

ZnO thin films with different number of layers/thicknesses were prepared using the sol-gel spin coating technique. First, the substrate used in this study, indium tin oxide (ITO), was cleaned by a standard cleaning process using ethanol and deionized water to eliminate all the contaminants and stains on the surface. Before the deposition process was conducted, the sol-gel ZnO solution was first prepared. Zinc acetate dihydrate, $Zn(CH_3CO_2)_2$ was used as the precursor. Monoethanolamine (MEA), C_2H_7NO, was added to the solution as the stabiliser, and 2-methoxy ethanol $C_3H_8O_2$, act as the solvent. 0.4M ZnO solution was stirred at 300 rpm, at 80 °C. After three hours, the heat was switched off, and the solution was continued to be stirred. The solution was aged for 24 hours to produce a clear and homogenous ZnO solution.

After 0.4M ZnO sol-gel solution was prepared, the deposition process was conducted via spin coating, using a spin coater (Laurell WS-650HZ-23NPP/UD2). The spin coater speed was set at 3000 rpm, with 60 seconds rotation. ITO substrate was placed at the center of the spin coater's stage, and when the rotation was started, ten drops of ZnO solution were dropped onto the substrate as the first layer. To produce ZnO thin films with a different number of layers (1, 3, and 5 layers), each of the layers undergoes the same process, which is spin-coated with ten drops of ZnO solution and dried at 150°C for ten minutes. To complete the fabrication process, deposited films were annealed at 400°C for one hour. Other deposition conditions, such as solution concentration, spin speed and duration, drying time and temperature, also annealing duration and temperature, were set to constant. The number of layers was increased before the samples were annealed, and each of the increased layers was dried before another layer was deposited.

The prepared ZnO thin films with a different number of layers were characterised by a few characterisations. The characterisations include field emission scanning electron microscope (FESEM, Hitachi SU-8030) to observe the thin films' morphology, and the surface profilometer (SP, KLA-Tencor P-6 Stylus Profiler) was used to determine the thickness of the different layers of ZnO thin films. Lastly, the crystalline quality of deposited ZnO films was analysed using x-ray diffraction (XRD, PANalytical X'Pert PRO). All of the obtained results were presented and discussed in the results and discussion section.

RESULTS AND DISCUSSION

As presented in the FESEM images of Figure 1 below, there is a significant difference in the ZnO thin films surface morphology for the thin film with 1-layer and 5-layers. For the 1-layer film in Figure 1(a), a uniform ZnO surface can be observed from the FESEM images, whereas the surface for the film with 5-layers shows a clear particle agglomeration (Figure 1(b)). Also, small cracks can be seen in Figure 1(a); however, in Figure 1(b), the film seems crack-free, although agglomeration can be observed. The explanation behind this occurrence might be due to the uniformity during the spin-coating process. For the 1-layer thin film, the distribution of the ZnO particles was not spread evenly onto the ITO substrate. Thus when

the number of layers was increased, it improves the uniformity of the ZnO particles. Based on the FESEM images provided, the structures of ZnO thin films improve significantly with the increment of the deposition layers. Dense and crack-free ZnO particles could be clearly observed in Figure 1 (b), indicating good quality of ZnO thin film. Findings obtained in this study were supported by the results reported by V. Kumar *et al.*, in which the morphology and structures of ZnO thin films also improved when the thickness of the thin film was increased [20]. V. Kumar *et al.* had varied the ZnO thin films' thickness by constantly depositing a 19-nm thin layer of ZnO films during each deposition layer and proven that the thicker film has better morphology. Besides, the structure of ZnO thin films deposited with 5-layer shows that the particles have a thicker thickness, correlated with the increase in the number of layers, which is expected and measured by the surface profiler.

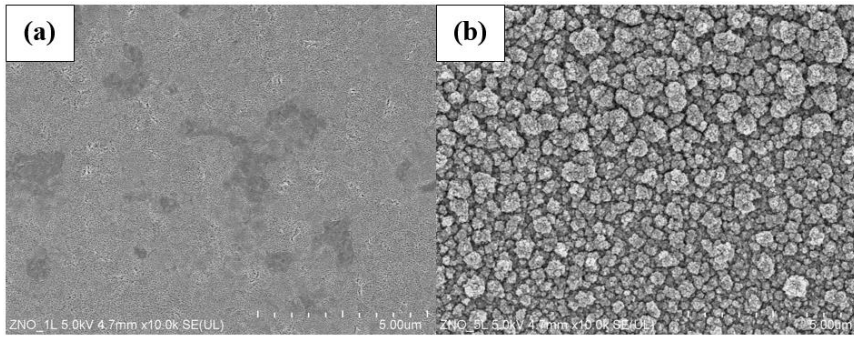

Figure 1: Morphology of ZnO Thin Film Deposited with (a) 1- Layer and (b) 5- Layers at 10K Magnification

Table 1 and Figure 2 below show the thickness value measured by the surface profilometer for all samples. The thickness value has a directly proportional relationship with the number of layers. As expected, when the number of layers was increased, significant changes in thickness can be observed. As shown in Table 1, for the thin film deposited with 1-layer, the thickness was below 100 nm, which is 58.52 nm. After further layers were deposited, the films become thicker. ZnO thin films deposited with 5-layers (highest number of layers) gave the highest thickness, with a value of 284.20 nm, almost five times the thickness of the 1-layer thin film. The thickness of the thin film increases due to more ZnO particles being deposited on top

of one another. S. S. Shariffudin *et al.* also obtained the same result trend when they varied the number of deposition layers for their ZnO thin films [21]. In their study, they varied the number of layers from 1-6 layers; thus, the thickness of the deposited ZnO thin films increases significantly with the number of layers and the grain size. Since the total thickness of the 5-layer thin film is almost five times the 1-layer thin film, we attribute the linear increment of the thickness to the drying process conducted at the end of each layer deposition. This drying process allowed the particles in one layer to be stable and dense before the subsequent layer was deposited. Thus, the number of particles increased with the addition of the layers increasing thickness [22]. The effect of the drying also was reported by M. Addamo [23]. In their study, M. Addamo *et al*. stated that when the deposited layer was dried layer by layer, each of the layers would grow over irregularly and preformed the crystalline surface or structure, which produced a film with thicker thickness [23].

Table 1: Thickness Value for all ZnO Thin Films Deposited with Different Layers

Number of layers	Thickness (nm)
1	58.52
3	195.03
5	284.20

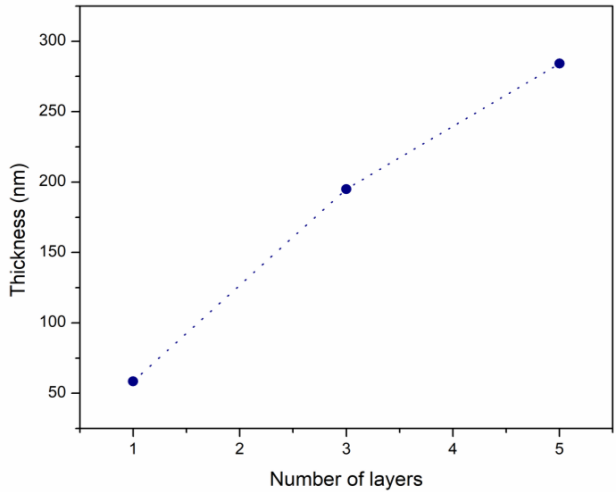

Figure 2: Comparison of Thickness Value for All ZnO Thin Films

To investigate the crystallinity quality of the deposited seed layer, all of the thin films were characterised using XRD. The XRD patterns for all of the thin films deposited with different layers are shown in Figure 3. As presented in Figure 3, ZnO thin film with 1-layer shows only a small peak of (101) orientation. As the thickness increases, significant changes in the crystallographic direction can be observed where a strong (102) peak can be seen for the 3-layer and 5-layer thin films. Also, small but clear (103) peaks can be seen for the thicker films. The improved intensity of (101) peak proportionally with the increase of film thickness might be due to the atomic layer that increased with the increment of the number of layers (thickness).

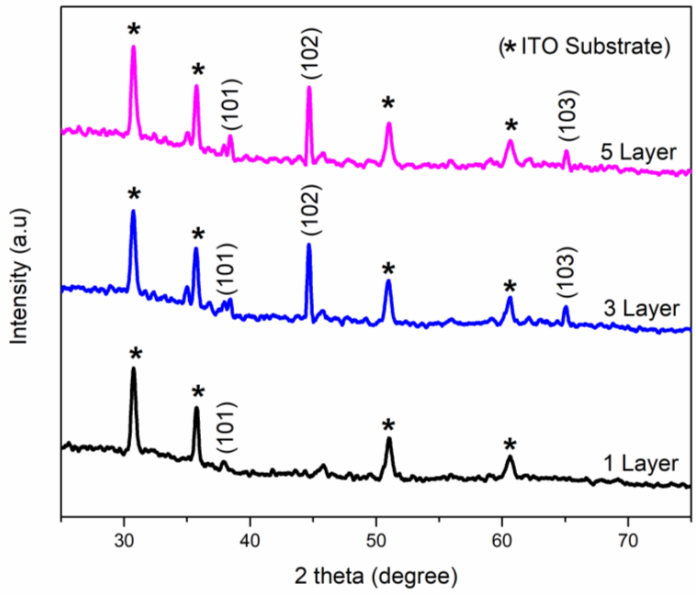

Figure 3: XRD Analysis for ZnO Thin Films Deposited with Different Layers

R. S. Goncalves *et al,* reported in their study that thicker film has more atomic layers to diffract x-rays [24]. This statement also had been supported by the findings by [25-27]. For the changes in the crystal orientation, we assume that as the layer increases, more atoms somehow tend to align in the (102) direction and pass the crystallographic information to the subsequent layers. Based on the XRD results, the mean crystallite size D, lattice constants a and c, and dislocation line density for all samples

were calculated based on the dominant peaks. Crystallite size, D, was calculated using Scherer's formula, as stated in Equation (1) below. While for dislocation line density, δ, lattice constants a and c, the calculations were calculated based on the formula in equations (2), (3), and (4), respectively.

$$D = \frac{0.94\lambda}{\beta \cos\theta} \tag{1}$$

Where D is crystallite size, λ is the source of wavelength, β is the diffraction line broadening at half of the maximum intensity (FWHM) of the peak, and θ is the peak position (obtained from 2θ).

$$\delta = \frac{1}{D^2} \tag{2}$$

For dislocation line density calculation, δ (Equation 2), D is the value of crystallite size, calculated using the formula in Equation (1). For lattice constants a and c, λ is the constant value of wavelength for x-ray radiation source (0.154 Å), while θ, is the value obtained from 2θ in XRD spectra. Both lattice constants a and c were calculated using Equation (3) and Equation (4) stated below:

$$a = \frac{\lambda}{\sqrt{3}\sin\theta} \tag{3}$$

$$c = \frac{\lambda}{\sin\theta} \tag{4}$$

Table 2 presents the crystallite size and other descriptions for peaks obtained from ZnO thin films deposited with different layers, analysed from XRD analysis. As presented in Table 2, crystallite sizes for all existing peaks increase as the number of layers increase. According to the data analysed, for the (101) peak, a significant increment of crystallite size could be observed, in which the value increase from 27.66 – 76.78 nm when further layers were deposited. The same trend was shown by (102) and (103) peaks, in which the crystallite size shows the highest value, proportionally with the thickness of ZnO thin films. Besides, by referring to the tubulated data, FWHM for all samples is inversely proportional to the crystallite size value. FWHM value showed a decrement trend when the number of layers was increased.

M. I. Khan *et al.* and A. Kamalianfar *et al*. also reported the same findings in their study [28-29]. The thickness of the thin film affects the crystallinity and crystallite size of the ZnO thin film. According to A. Kamalianfar, the thicker film would give better crystalline quality because thicker film tends to have a smaller value of δ (dislocation line density) [29]. This statement was supported by the findings obtained in this study, in which the value for δ in this study decrease as the number of layers increases. A smaller value of δ means that the film has less atom arrangement dislocation, thus improving crystallinity quality.

Table 2: The Relative Orientation, 2 Theta (2θ), FWHM, Crystallite Size, Dislocation Line Density, and Lattice Constants Value for ZnO Thin Films with Different Number of Layers

Number of layers	Orientation, hkl	2 theta (2θ)	FWHM	Crystallite size, D (nm)	Dislocation line density, δ ($\delta \times 10^{-3}$ line/nm^2)	Lattice constants	
						a	c
1	101	37.84	0.5341	27.66	1.307	2.742	4.749
3	101	38.06	0.3245	45.52	0.483	2.727	4.723
	102	44.55	0.2838	52.06	0.369	2.346	4.063
	103	64.99	0.3147	46.96	0.437	1.655	2.867
5	101	38.48	0.1885	76.78	0.169	2.698	4.673
	102	44.68	0.2787	53.02	0.356	2.339	4.052
	103	65.12	0.3059	48.31	0.425	1.652	2.861

The decrement of dislocation line density can also be linked to the crystallographic defect in the materials used. Referring to this statement, δ value could determine the defects of the materials, thus smaller δ value, less defect in crystallographic. Besides, M. I. Khan stated that if the crystallite is larger, more atoms will be in order; thus, there will be fewer grain boundaries [28]. Therefore, atoms will easily move from grain to grain due to the fewer grain boundaries. Consequently, from this effect, the electrical properties of deposited ZnO thin films will be improved. As for the lattice constants, a and c, the value for all peak's orientation decrease with the increasing thickness of ZnO thin films. Data presented in Table 2 showed a slight decrement of lattice constants value when further layers were deposited. This decrement of lattice constant might be due to the crystallite size, which

increases when the thickness increase. This increment will cause the lattice restoration, hence influencing the lattice constants' value [30].

CONCLUSION

In summary, ZnO thin films with the different number of layers, prepared using the sol-gel spin coating technique had been presented in this study. The denser and crack-free structure was obtained by the 5-layers sample, as observed by FESEM. The thickness of the sample was also affected, which 5-layers ZnO thin films increase almost five times (289 nm) compared with the 1-layer sample (58nm). This increment had improved the crystalline quality of the ZnO thin films, as the intensity of (101), (102), and (103) peaks increase. Also, the crystallite size had increased with the increment of the film thickness with the range from 27 ~ 77 nm. Besides, the decrease of dislocation line density, δ and lattice constants (a and c) shows that thicker film has better crystalline quality with less defect crystallographic. Based on these findings, ZnO thin films deposited with 5-layers can be a suitable candidate to be the seed layer to grow the zinc oxide nanorod (ZNR). The grown ZNR could be used as the n-type material in solar cell application because ZNR can improve the efficiency of a solar cell by increasing the amount of light trapping during the measurement process.

ACKNOWLEDGEMENT

The authors would like to thank all members of Nano Electronic Center (NET), Integrated Sensor Research Group (DERIA), and Nano-Science Technology Center (NST), Universiti Teknologi MARA (UiTM) Shah Alam for all the research facilities. This work is partially supported by the Ministry of Higher Education Malaysia under the *Fundamental Research Grant Scheme* (600-IRMI/FRGS 5/3 (080/2017)).

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