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Tailoring of ZnO thin films: effect of number of coating and sample ageing

Abstract. In this work, the effect of number of coating and sample ageing on the properties of ZnO thin films prepared by dip coating technique using 0.1 M aqueous solution of zinc acetate and methanol on the glass substrate was experimentally investigated. The linear four probe configuration has been used to measure sheet resistance of the deposited samples whereas laboratory purpose Hall-effect measurement apparatus has been used to measure carrier concentration and Hall mobility. The dark sheet resistance (180.37 kΩ/□ to 41.69 kΩ/□) and normal sheet resistance (163.15 kΩ/□ to 31.72 kΩ/□) of ZnO thin films decreases significantly as the sample age increased from 79 to 167 days, particularly for films deposited with 8, 12, 16, and 20 coats. It is also found that as the number of coating increases, thickness of the thin films (from 199.7 nm to 211.5 nm) and carrier concentration increases whereas the Hall mobility decrease. The spectrophotometer (USB2000, photonics) has been used to investigate optical properties of deposited films. All the ZnO thin films demonstrate remarkable transparency, exhibiting transmittance lies between 61% and 85% across the wavelength range of 450 nm to 950 nm. The optical transmittance is observed to decrease with increase in number of coatings in the wavelength range 650 to 950 nm. The optical band gap value increases slightly from 3.22 eV to 3.25 eV for corresponding number of coatings from 8 to 20. This slight change in optical band gap indicates that it does not depend significantly on number of coating and thickness of the films.
Key words: carrier concentration, dip coating technique, hall mobility, optical band gap, zinc oxide thin films.

Introduction

Zinc Oxide (ZnO) thin film is one of the pioneering materials lying into II-VI group compound semiconductor [1, 2]. It is an oxide semiconductor with a wide and a direct energy gap of around 3.37 eV. ZnO has high mechanical and chemical stability as well as high abundance [3]. Thin films of ZnO are technologically significant due to their wide range of electrical and optical properties which make them appropriate for number of applications in solar cells, gas sensor, optical waveguide etc. [4-9]. The thin films produced using the ultrasonic spray pyrolysis technique has explored the structural, morphological, photoluminescent, photocatalytic and photocurrent characteristics of ZnO thin films, studying how these properties vary in relation to the thickness of the film ZnO [10]. Zinc oxide is less toxic and more affordable than its competitors, in addition to possessing good electrical and optical properties [11, 12]. ZnO thin films show n-type semiconductor behavior where its conductivity and resistivity values can be controlled by deposition technique, preparation conditions and dopant source [13]. The variety of techniques including magnetron sputtering [14, 15], atomic layer deposition [16], sol-gel technique [17-20], electrochemical deposition [21], wet method with water-soluble polymers [22], chemical vapor deposition [23], pulsed laser deposition (PLD) technique [24-26], spray pyrolysis [27] etc. are known. Of these, the sol-gel process is an environmentally favorable [28] and simple method that is used to prepare thin films of ZnO on a large surface [29, 30]. Therefore, it is very easy to implement at industrial level to get the thin films of good quality. Generally, the sol-gel approaches use three methods and they are dip-coating, spin-coating and spray coating. Recently, Yadav et al., [1], have extensively studied the effect of solution age, sample age and aluminum concentration on the optical and electrical properties of ZnO:Al films. However, they have not reported the effect of sample age on sheet resistance for pure ZnO thin films deposited with different number of coatings of solution age 10 days. Gonzalez, and Urueta [31] studied the variation of dark and photo-resistivity with the film thickness in both pure and aluminum doped ZnO films. They observed that when film thickness increases, resistivity decreases systematically in aluminum–doped ZnO thin films prepared with the sol-gel technique but in pure ZnO thin films, there was no such systematic variation in resistivity with film thickness.

These facts motivated us to study the systematic variation of sheet resistance, carrier concentration, Hall mobility with the different number of coatings, sample age and film thickness. In this work, considering the above motivation, we have studied the effects of number of coating and thickness on electrical properties (both in dark and normal light condition) and optical properties of ZnO thin films of 0.1 M solution deposited on to glass substrate using the dip coating method.

Materials and Methods

Sol-gel process refers to the change of liquid “Sol" to solid "Gel" phase. A net "Gel" will form when "Sol" is cast into a mold. The "Gel" is transformed into dense ceramic or glass articles after drying and heat treatment. In this way, such thin films can be prepared on a glass substrate using the dip coating technique.

In order to deposit ZnO thin films, standard disposable microscopic glass substrate having the length = 75±1 mm, breadth = 25±1 mm, thickness = 1.35±0.1 mm was cleaned chemically and ultrasonically for 15 minutes using an ultrasonic cleaner (PS-60A, ROHS). It was then allowed to dry for 5 hours at room temperature in an oven prior to employing the dip coating process. For the deposition of homogeneous solution of ZnO, 0.1 M solution of zinc acetate [$Zn(CH_3COO)_2$] (Ava Chemicals, India, molecular weight 219.50 gm, minimum assay 99.5%) as a precursor and pure methanol (CH_3OH) (Sisco Research Laboratories, India, molecular weight 32.04 gm, minimum assay 99%) as solvent was mixed and stirred using magnetic stirrer. The following chemical reaction given below results the production and formation of ZnO thin film on the glass substrate:

$$Zn(CH_3COO)_2 \rightarrow ZnO + CO_2\uparrow + CH_3COCH_3\uparrow$$

Acetone (CH_3COCH_3) evolved here was a volatile substance which was evaporated by heating and layer of ZnO was formed on glass substrate. Before deposition, the prepared solution was given 10 days to age. A glass substrate that had been cleaned was dipped in a 100 ml borosilicate glass beaker containing a 0.1 M solution and it was then withdrawn from solution slowly by substrate pulling system. The deposition rate was altered by changing the withdrawing velocity. For this instance, a withdrawal velocity of 0.2 cm/s was selected as the standard. After deposition, the solution was allowed to evaporate at 50 °C, and the zinc oxide film was allowed to crystallize at constant temperature 490 °C by keeping the substrate over heater and it was then taken out from heater to cool down at room temperature. To increase a greater number of coatings and thickness of the films, this process (dip coating technique) was used multiple times i. e. 8, 12, 16, and 20 times respectively to fabricate thin films of ZnO. By using this approach, the transparent, uniform and smooth thin films of ZnO on the glass substrates were produced.

For sheet resistance measurement, linear four probe configuration was used. In addition to this, laboratory purpose Hall-effect measurement apparatus was used for the carrier concentration measurement and mobility determination. To assess the optical properties i. e. transmittance was measured in the wavelength range of 367 to 1038 nm using a spectrophotometer (USB2000, photonics). The absorbance and band gap energy values of the deposited ZnO thin films were also calculated using the transmittance data.

Electrical properties

To perform electrical characterization, the relation provided below is utilized to determine sheet resistance [1].

$$\text{Sheet resistance } (Rs) = \frac{\pi}{ln\,2}\frac{V}{I}$$

In this process, the measurement of the voltage V was carried out between the two inner probes through the implementation of the four-probe method in a linear configuration while the current I was measured through the two outer probes.

For study of effect of number of coatings on carrier concentration, we have used the relation given below which determined the carrier concentration [1] values of samples deposited with varying number of coatings.

$$n = -\frac{1}{(R_H \times e)}$$

Where R_H is Hall coefficient, e is charge of electron. As the Hall coefficient (R_H) was determined through the following expression [1].

$$R_H = \frac{d}{B}\left[\frac{R_{31.42} + R_{42.31}}{2}\right]$$

Where, $\left[R_{31,42} = \frac{V_{42}}{I_{31}}\right]$. Also d is the thickness of the film and B is the applied magnetic field. To carry out this process, a current I was supplied through the two probes 1 and 3, while the voltage V generated across the other two probes, namely 2 and 4, was measured. The thickness *(d)* of the ZnO films was determined from interference fringes of transmittance data by optical method.

In addition to that we have measured the value of Hall mobility *(μ)* of the charge carriers with known electrical resistivity and Hall coefficient with different number of coatings by using the relation,

$$\mu = \frac{R_H}{\rho}$$

Where, resistivity *(ρ)* of ZnO thin films was determined by multiplying sheet resistance *(Rs)* and film thickness *(d)* [32, 33].

Optical properties

To explain the behavior of optical band gap with number of coatings, the Tauc relation has been utilized to determine the optical band gap from the measured transmittance.

$$\alpha = \frac{A}{h\nu}\left(h\nu - E_g\right)^{1/2}$$

The expression $A = ln\left(\frac{100}{T}\right)$, where T is transmittance, is referred to as optical absorbance. By plotting $(\alpha n \nu)^2$ versus hν, the possible transition occurring and corresponding energy gap for the material was found [20].

Results and Discussion

Effect of Number of Coating on sheet resistance and thickness of thin films

To study the effect of number of coatings, ZnO films on glass substrate with varied coating numbers of 8, 12, 16, and 20 were prepared in the same day after 10 days ageing time of starting solution. The ordinary light condition was set up from electric lamp and the illumination condition is referred to the stage of measurement of characteristics. Figure 1 represents the variation of normal (in the presence of ordinary light from electric lamp) and dark (in absence of light) sheet resistance (*Rs*) of ZnO films with varying number of coatings keeping sample age 79 days. It can be observed from the curve that the normal sheet resistance is lower than the dark sheet resistance that was measured under the same time and temperature conditions. Gonzalez and Urueta have also reported a similar observation [31]. It is evident that the sheet resistance of ZnO films decreases systematically as the number of coatings increases. The dark sheet resistance decreases from 180.37 kilo-ohm/square (kΩ/□) to 41.69 kΩ/□ when number of coating increases from 8 to 20 coat whereas normal sheet resistance decreases from 163.15 kΩ/□ to 31.72 kΩ/□. This shows that dark and normal sheet resistance have similar trend. Kandpal et al. have also reported a comparable observation [33].

It has been known that the sheet resistance of ZnO film is determined by two interdependent factors: the stoichiometry of ZnO and the degree of non-stoichiometry of ZnO. The stoichiometry of ZnO film can be altered by either substituting Zn^{2+} or by introducing oxygen vacancies. As the number of oxygen vacancies increases, the sheet resistance decreases [34, 35].

The thickness of the ZnO thin films increases gradually from 199.7 nm to 211.5 nm (8 coating – 199.7 nm, 12 coating – 201.7 nm, 16 coating – 206.5 nm, 20 coating – 211.5 nm) with increase in number of coatings from 8 to 20 coat. It can be noticed that the thickness per dipping for the first dip is lower than for the succeeding dips. This is because the initial deposit is made on a glass substrate and the following deposits are made directly on the crystalline ZnO layer itself. Ray and Karanjai obtained the similar results for dip-deposited SnS and SnS_2 thin films [36, 37].

In addition, we have measured sheet resistance of same samples after 167 days from the date of preparation. Figure 2 indicates the sheet resistance variation with number of coating and these show similar trend for normal and dark conditions. However drastic change in sheet resistance is found as the sample aging increases from 79 days to 167 days. It is found that the sample with 20 coat has normal sheet resistance 31.72 kΩ/□ when measured after 79 days from the date of sample preparation whereas after 167 days, the normal sheet resistance is found to be 285.71 Ω/□. The value of dark sheet resistance for 167 days sample age is also smaller in comparison to the value of sheet resistance for 79 days sample age. This is due the different sample ageing-time [31, 38]. So, it is observed that sheet resistance decreases as sample ageing increases which we discussed earlier in our previous work for ZnO:Al thin films [1].

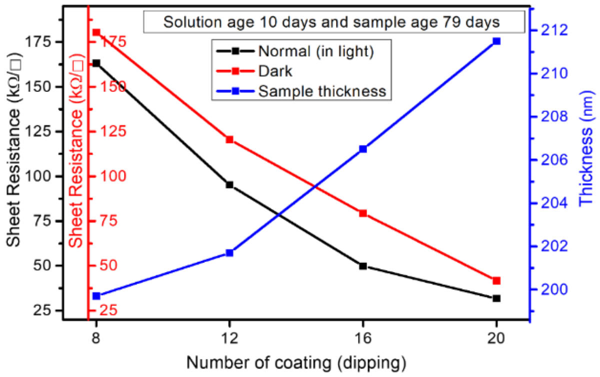

Figure 1 – Normal and dark sheet resistance vs thickness and coating number of 79 days age ZnO films synthesized using 10 days age solution.

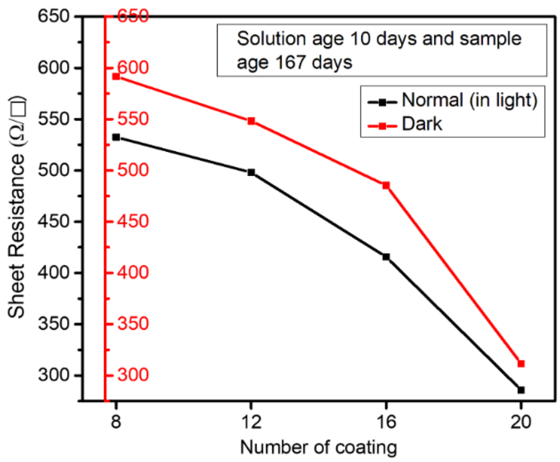

Figure 2 – Normal and dark sheet resistance vs coating number of 167 days sample age of ZnO films synthesized using 10 days age solution

The decrease in sheet resistance with increase in number of coating can also be mentioned on the fact of increase in carrier concentration. The observed decrease in sheet resistance with increasing number of coatings can be attributed to the corresponding increase in carrier concentration in the ZnO thin films, as depicted in Figure 3. As the ZnO films are heated, evaporation of oxygen takes place which results in the formation of oxygen vacancies. These vacancies are then promptly ionized by releasing two electrons in the conduction band. Thus in turn carrier concentration increases and sheet resistance of ZnO thin films decreases [39].

Effect of number of coatings on carrier concentration and Hall mobility

Figure 3 shows the plotting of carrier concentration and Hall mobility of thin films of ZnO with varying numbers of coatings which is used to elucidate the behavior of carrier concentration and Hall mobility with respect to the number of coatings. The plot in Figure 3 displays that as number of coating increases, carrier concentration of ZnO thin films also increases, however, Hall mobility decreases. The decrease in Hall mobility of the ZnO film is attributed to the chemisorption of oxygen at the grain boundaries, leading to the formation of extrinsic trap states that are localized at the grain borders. These extrinsic trap states at the grain boundaries trap free carriers from the bulk of the grains and create potential barriers, leading to depletion in regions adjacent to the grain boundaries. As a result, the mobility of the carriers is reduced [40].

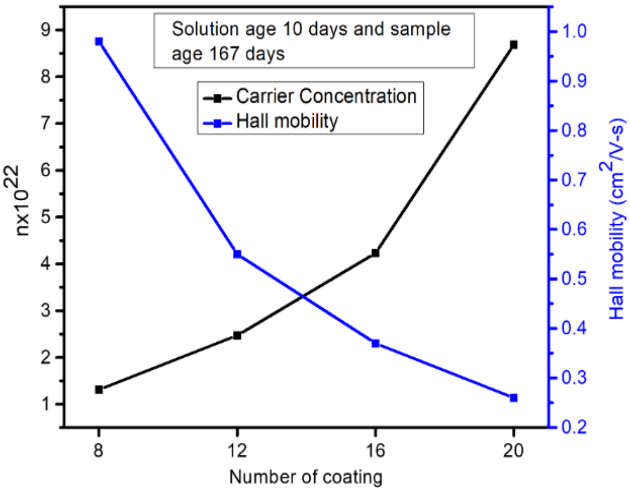

Figure 3 – Carrier concentration and Hall mobility variation with different number of coatings for ZnO films of solution age 10 days and sample age 167 days

Optical properties

Figure 4 displays how the optical transmittance changes across the wavelength range of 382 nm to 1025 nm for ZnO films prepared using a solution aged for 10 days and coated with different numbers of layers, including 8, 12, 16, and 20 coatings. The curves in plot indicate that all the prepared samples exhibit high transparency. The films show transmittance values ranging from 61% to 85% in the wavelength range of 450 nm to 950 nm. The transmittance spectra show superimposed optical interference, indicating the films' good homogeneity [31]. It can be observed that transmittance sharply decreases below threshold wavelength of about 382 nm. The films' transmittance decreases in the wavelength range of 650 nm to 950 nm as the number of coatings (thickness) increases [35, 41-43].

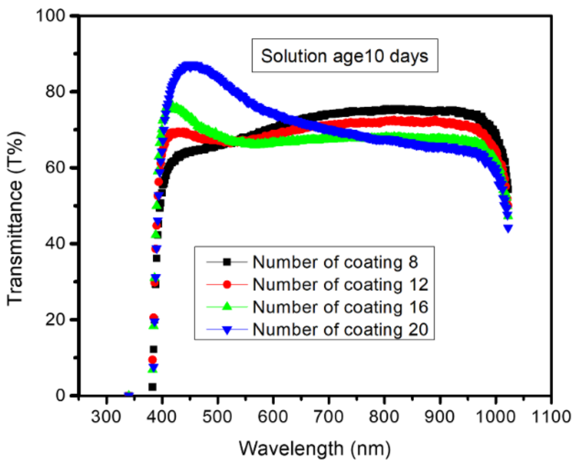

Figure 4 – Variation of transmittance with wavelength for deposited ZnO film with 10 days aged solution

Figure 5 illustrates the changes in absorbance with wavelength for ZnO films that were made using 10 days aged solution and varying numbers of coatings. From the curve, it is observed that absorbance sharply decreases with wavelength. The spectra show sharp cut off and is obtained around the wavelength 384 nm and beyond this value i.e. in the range of wavelength from 450 nm to 1000 nm, absorbance is very low and nearly constant for all samples. When wavelength, λ>1000 nm, the energy of the incident photons is not sufficient to interact with atoms [44] and hence the influence of interference effect may be excluded.

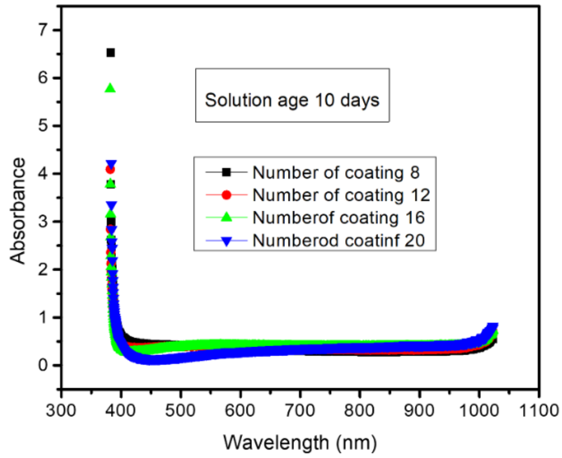

Figure 5 – Variation of absorbance with wavelength of ZnO thin film deposited with 10 days aged solution.

Figures 6(a), 6(b), 6(c) and 6(d) display the plots of $(\alpha h\nu)^2$ versus $h\nu$ for the ZnO thin films with varying numbers of coatings, specifically 8, 12, 16, and 20 coats respectively.

The band gap of the ZnO thin films was determined by performing linear fitting of the straight-line portion of the curve. The band gap of samples with number of coatings 8, 12, 16 and 20 are found to be 3.22 eV, 3.23 eV, 3.24 eV and 3.25 eV respectively. The enhancement of band gap in the thin films of ZnO deposited with different number of coating and thickness is shown in Figure 7. This study reveals minimal changes in the optical band gap of ZnO films when the number of coatings and film thickness are varied. Such a small increase in band gap is due to the growth of larger grain size as the film thickness increases [3]. However, this finding implies that the band gap of ZnO films is not substantially influenced in significant number by either the number of coatings or the thickness [3, 45, 46]. Ennaceri et al. have also reported a similar observation [35] for thin films of ZnO deposited on glass substrates using ion laser gas reaction process.

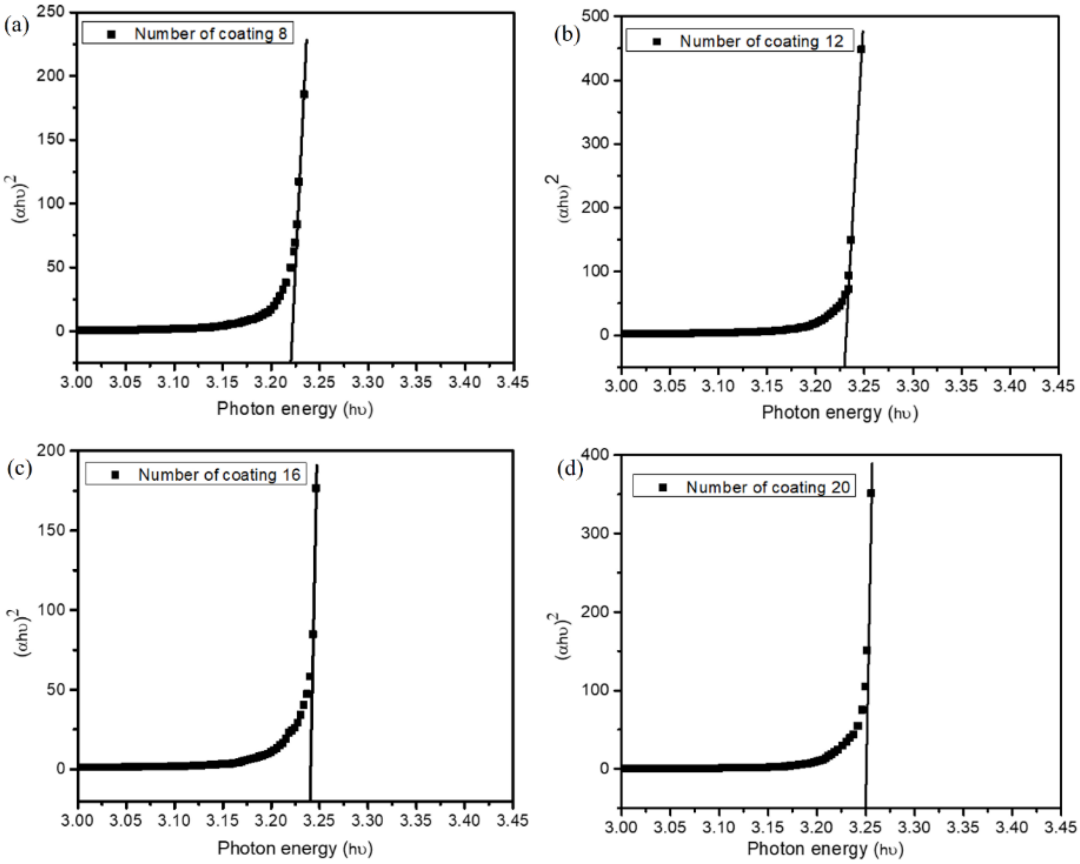

Figure 6 – Band gap variation with (a) 8 number of coating (b) 12 number of coating (c) 16 number of coating and (d) 20 number of coatings of ZnO films deposited with 10 days aged solution.

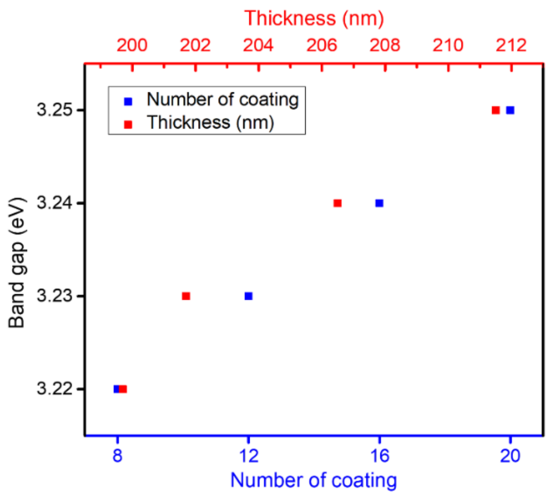

Figure 7 – Band gap variation with number of coating and thickness of ZnO films of 10 days aged solution

Conclusion

Using a low-cost dip coating process, transparent and conductive undoped ZnO thin films were prepared on glass substrates. The study presented the influence of the number of coatings on various properties of the films, including electrical sheet resistance, film thickness, carrier concentration, Hall mobility, and optical properties. It was observed that the sheet resistance of the ZnO thin films, which were deposited with 8, 12, 16, and 20 coats, exhibited a significant decrease as the sample age increased from 79 days to 167 days. It was also noted that an increase in the number of coatings simultaneously leads to an increase in the thickness of the thin films. Moreover, the study revealed that as the number of coatings increased, the carrier concentration of the thin film increased, while the mobility of the charge carriers decreased. In addition, it was observed that all the ZnO thin films exhibited high transparency, with transmittance ranging from 61% to 85% in the wavelength range of 450 nm to 950 nm. Furthermore, the optical transmittance was noted to decrease as the number of coatings increased within the wavelength range of 650 nm to 950 nm. Interestingly, the finding of this work suggests that neither the number of coatings nor the thickness has a significant impact on the changing band gap of ZnO films.

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