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Ceramics International 39 (2013) 5681–5687

CERAMICS INTERNATIONAL

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Low power deposition of high quality hexagonal ZnO film grown on Al_2O_3 (0001) sapphire by dc sputtering

K. Vijayalakshmi[a,*], K. Karthick[a], P. Dhivya[b], M. Sridharan[b]

[a] *PG and Research Department of Physics, Bishop Heber College, Tiruchirapalli 620017, India*
[b] *Functional Nanomaterials and Devices Lab, Centre for Nanotechnology and Advanced Biomaterials and School of Electrical and Electronics Engineering, SASTRA University, Thanjavur 613401, India*

Received 27 November 2012; received in revised form 25 December 2012; accepted 25 December 2012
Available online 11 January 2013

Abstract

High quality hexagonal block of nanostructured zinc oxide (ZnO) thin films were grown on Al_2O_3 (0001) sapphire substrate at room temperature by direct current (dc) magnetron sputtering, with low sputtering power from 10 to 50 W. The effect of sputtering power on the structural, optical and morphological properties of the films was investigated by X-ray diffraction (XRD), photoluminescence spectrometer, UV–vis spectrometer and scanning electron microscope (SEM), respectively. From the XRD pattern, it is inferred that (002) oriented peaks is present in all the samples prepared under various sputtering powers. The presence of a strong single UV emission at 385 nm is attributed to the near band edge emission, confirming that the film have less defect density signifying the high crystal quality of the samples. The films were highly transparent with sharp absorbance edges, and average visible transmittance is of 90%. SEM micrographs revealed that the grains become more uniform with very little mismatch with the surface of c-cut sapphire substrate, with increase of sputtering power. The film prepared at the dc power of 40 W results in a layer containing well shaped hexagonal wurtzite ZnO.

Keywords: ZnO nanoblock; Sputtering; Photoluminescence; SEM

1. Introduction

Materials are considered to have wide bandgap, if their bandgap energy is significantly greater than that of common semiconductors like silicon and gallium arsenide (1.4 eV). Wider bandgap materials like zinc oxide (ZnO) is more efficient in high power and high temperature applications, where the materials limit of silicon based device are being reached. ZnO is the focus of this study and, it is a semiconducting material with a bandgap of $\sim$3.3–3.4 eV and a non-centrosymmetric hexagonal wurtzite crystal structure [1,2]. It can withstand higher powers owing to the higher breakdown voltage associated with higher bandgap. In the past two decades, nanostructured ZnO have drawn significant attention due to their structural and electronic properties originating from their large surface to volume ratio. The optical properties of ZnO are strongly influenced by the confinement of electrons and holes. The sum of kinetic and potential energy of the freely moving carrier is responsible for the energy gap expansion, and therefore the width of the confined energy grows as the characteristic dimension of the crystallite decreases [3]. Moreover, the nature of defects in a semiconductor material corresponds to its optical property. Due to radiative recombination, ZnO finds its most use in optoelectronics. Above all the complete use of ZnO in optoelectronics application guides well to understand the properties of ZnO, that leads to the growing number of device applications.

Various substrates and deposition techniques have been employed in an attempt of achieving high quality growth, defect less structure for device development. Among several kinds of substrates, sapphire remains the most common choice for heteroepitaxy despite its poor structural and thermal mismatch with metal oxide films resulting in a considerable strain after fabrication of the films [4]. In the

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[*] Corresponding author. Tel.: +91 99 9464 7287.
E-mail address: viji71naveen@yahoo.com (K. Vijayalakshmi).

http://dx.doi.org/10.1016/j.ceramint.2012.12.085

past decades, several methods have been developed to prepare ZnO thin films including sputter deposition, vacuum coating technique, solvothermal, spray pyrolysis and sol–gel method [5–9]. In the present work direct current (dc) magnetron sputtering have been used for the preparation of ZnO thin films for its advantages; low substrate temperature (down to room temperature), good adhesion of films on substrates, high deposition rates, good thickness uniformity and high density, good controllability and long term stability of the process and easy fabrication of metal oxide films with different parameters [10]. Along with other parameters, the sputtering power is perhaps the most critical parameters determining the structure and properties of ZnO films. Moreover, high temperature deposition has the disadvantage of producing degradation of the substrate, incorporation of impurities and thermal damage to the growing films [11]. Hence, the deposition of ZnO films at low temperature has become increasingly important and value. Therefore to optimize the films for different dc power during deposition, in order to get the reproducible films, a study on the growth of ZnO thin films at room temperature has been carried out and the effect of dc power on the structural, optical and photoluminescence properties of the films were investigated.

2. Experimental details

ZnO thin films were deposited on c-plane sapphire (0001 plane) substrates by dc magnetron sputtering using the zinc (99.99% purity Sigma Aldrich) metal target of 2 in. diameter. The sapphire substrates were cleaned in successive baths of acetone, ethanol and deionized water for 10 min at room temperature. The cleaned substrate was loaded in the sputtering chamber with a target to substrate distance fixed at 4 cm. The chamber was evacuated to the base pressure of about 10^{-5} Pa, and the deposition pressure was maintained at 10^{-1} Pa. Pre-sputtering was carried out for 15 min to remove the contaminations on the target before each deposition. The ZnO thin film was sputtered with gas mixture of high purity O_2 and Ar in the ratio of 1:2 at a constant deposition time of 30 min. The substrate was kept at room temperature and the sputtering power was changed as 10, 20, 30, 40 and 50 W to study the effect of dc sputtering power on structural, optical and photoluminescence properties of the films. Fig. 1 shows the schematic of the experimental set-up used for the deposition of ZnO thin films by dc sputtering method. The crystalline structure and the orientation of the ZnO films were examined by an X Perto X-ray diffractometer with CuKα radiation. Room temperature PL studies were carried out using a Varian Carry Eclipse fluorescence spectrophotometer with a Xenon flash lamp technology. The transmission of the film was measured with Perkin-Elmer-UV–vis Lambda 35 Spectrometer, and SEM analysis of the ZnO films were carried out using a HITACHI high technology SU9000 UHRE field emission scanning electron microscope.

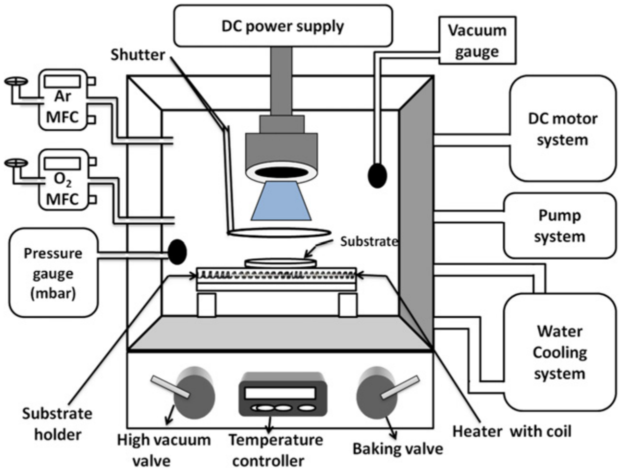

Fig. 1. Schematic of the experimental set-up used for the deposition of ZnO thin films by dc sputtering method.

3. Results and discussion

3.1. Effect of sputtering power on the lattice parameters of ZnO films

XRD patterns of the ZnO films deposited on c-plane (0001) sapphire substrate at different sputtering power are shown in Fig. 2. The strongest (002) preferred c-axis oriented peak was observed for all the samples around 34.42°, indicating that the films are crystallized in the wurtzite phase structure. The intensity of (002) peak relative to the background demonstrates the purity of the hexagonal Zn phase and the crystallinity of the samples. From the pattern it is clearly seen that, no extra peak other than (002) peak from ZnO and (0001) peak from sapphire was found from the X-ray diffraction scans between 30 and 50 degrees. All diffraction peaks are indexed with JCPDS data sheet 63-1451. Moreover, it was observed that for the film deposited at a dc power of 10 W, the XRD peak intensity is low, because of the low crystalline growth of the films on substrate, due to insufficient kinetic energy of the sputtered ZnO atoms and consequently the reduction in the mobility of adatoms to a very low value. The intensity of the peak increased with increasing sputtering power upto 40 W, and then decreased when the power was further increased to 50 W. The peak intensity was found to be high for the samples prepared at 40 W indicating a better crystallinity. So, the optimum dc power to obtain uniform well adherent ZnO film was found to be 40 W.

The effect of sputtering power is to supply kinetic energy to the sputtered particles [12]. As a result, the crystallinity and the donor activation by substitution will be increased. However its benefits on improved mobility are effective only at room temperature deposition, since the thermal energy contribution is low at that time. From this, it is inferred that when the sputtering power raises, the energy or number of electrons and ions in the glow discharge plasma will increase. The positive ions gain higher energy because of the raising sputtering power and they will strike

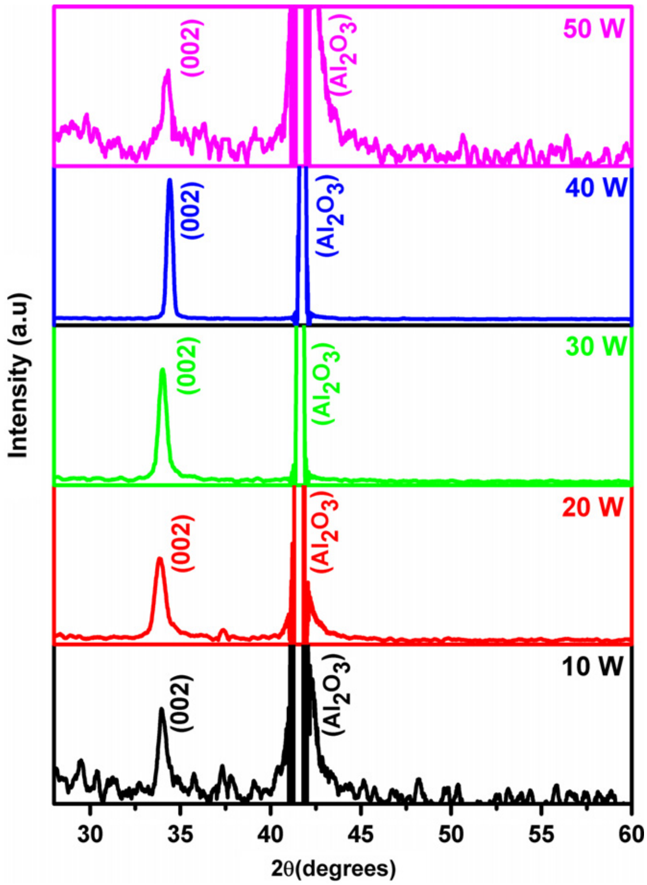

Fig. 2. X-ray diffraction patterns of the ZnO film prepared for different sputtering power.

the target. Thus, with an increase in sputtering power, the sputtered atom receives high energy from the positive ions as a result of collision. This high energy can be used to form a patterned crystalline structure thereby improvising the crystallinity in the film. However, the excessive supply of sputtering power (50 W) may cause a degradation of the (002) preferred orientation, resulting in the high defect density. This implies the increase in dislocation, interstitials or vacancies and the chance to miss the most energetically favored sites when the sputtering power is increased. The mean crystallite size D of ZnO film deposited at different sputtering power was estimated by the Debye-Scherrer's formula [13]:

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

where k is the shape factor of the average crystallite (0.91), λ is the wavelength of the incident X-ray ($\lambda = 1.5418\pi^0$), β is the full width at half-maximum (FWHM) in radians and θ is the Bragg angle. Fig. 3 shows the variation of FWHM and crystallite size of ZnO (002) diffraction peak as a function of sputtering power. The value of FWHM increased firstly and then decreased with the increase of sputtering power, which also indicates the trend of the crystal quality of ZnO films. For the films prepared at 40 W, the FWHM was found to be 0.268° far narrower than that reported previously ($>0.5°$) [14], indicating the better crystalline quality of the deposited ZnO films. At the power of 50 W, FWHM again increased, indicating the deterioration of the crystalline quality.

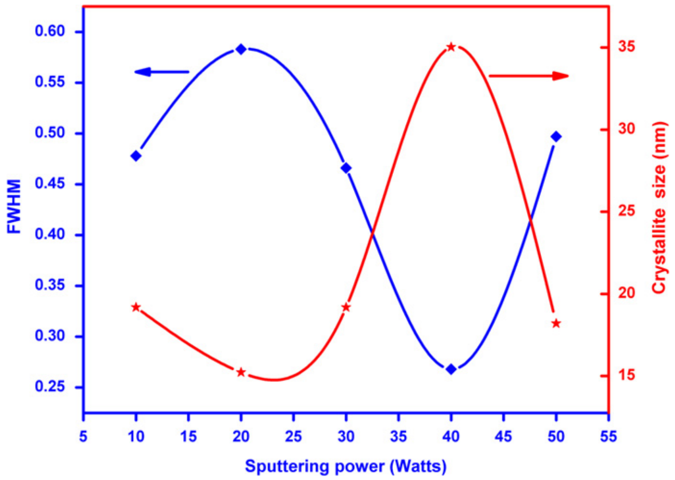

Fig. 3. Variation of FWHM and crystallite size of ZnO films as a function of sputtering power.

Moreover, the dependence of crystallite grain size of the film on dc power may be attributed to the lattice mismatch between the grown ZnO film and the c-plane (0001) sapphire substrate. The lattice constant 'a' and 'c' were calculated using the following relation [15]:

$$\frac{1}{d^2} = \frac{4}{3}\left[\frac{h^2+hk+k^2}{a^2}\right] + \frac{l^2}{c^2} \tag{2}$$

The lattice constant 'a' and 'c' values were found to be larger than that of bulk ZnO of 3.249 Å and 5.206 Å. This indicates that the films have residual strain and stress along c-axis. In the interface of nanomaterials, lattice strain on the surface is one of the prominently basic and essential qualities [16]. This property is widely used in various domains, since it suggests new properties when compared to the bulk counterparts. Using the lattice strain, the optical, electronic and mechanical characteristics of nanoparticles can be efficiently tuned. The values of c-axis strain (ε_z) was calculated by using the formula given below [17]:

$$\varepsilon_z = \frac{c_{film} - c_{bulk}}{c_{bulk}} \times 100\ \% \tag{3}$$

where c_{film} is the lattice parameter of the strained ZnO film calculated from X-ray diffraction data and c_{bulk} is the unstrained lattice parameter of ZnO. The compression or tension stress of nanoscale materials can modulate the lattice parameters. The stress in the plane of the films was calculated based on the biaxial strain model, using the following formula [18]:

$$\sigma_{film} = \frac{2C_{13}^2 - C_{33}(C_{11} - C_{12})}{C_{13}} \times \varepsilon_z \tag{4}$$

where $C_{11} = 209.7$ GPa, $C_{12} = 121.1$ GPa, $C_{13} = 105.1$ GPa, and $C_{33} = 210.9$ GPa are the elastic stiffness constant of bulk

ZnO. ε_z is the strain of the deposited film. When it comes to thin films, the intrinsic and extrinsic stresses contribute to the total stress [19]. During thin film deposition, intrinsic stress arises owing to defects and impurities, since films are deposited under non-equilibrium conditions. In addition, the thermal expansion coefficient mismatch and lattice mismatch between the film and the substrate can be linked with extrinsic stress. In accordance to the stress equation, biaxial stress is tensile if stress value is positive and compressive if the stress is negative [20]. In the present case, intrinsic stress is completely dominant and extrinsic stress is almost absent. Fig. 4 shows the variation of tensile stress and the residual strain in ZnO film for different power. At the power of 20 and 30 W, the diffusion rate of ZnO atoms to the surface of the substrate is less, which revealed that the stress and strain is high. At the power of 40 W, the strain and tensile stress is much minimum, which may be due to the variation in crystallite size, decrease in vacancies, dislocation, intrinsic defects, and the sputtered atoms would have selected the favored position on the surface of the substrate. At 50 W, the values increased slightly indicative of possible effect of lattice distortion and defects in the film. The observed XRD pattern of our samples with high intensity unique *c*-axis orientation and less stress prepared at low sputtering power and low temperature are reasonably superior to that reported by Rambu et al. [21] on Al_2O_3 sapphire at high temperature.

3.2. *Effect of sputtering power on the light emission characteristics of ZnO films*

PL spectrum is essential to analyze the extrinsic and intrinsic properties of semiconductor [22]. The PL technique can provide valuable information on the detection of defects and the recombination process in semiconductors. The luminescence property of the deposited films for different sputtering power is depicted in Fig. 5. From the emission spectra, it is inferred that the near band emission (NBE) in the UV region is at around 385 nm. The previous reports on PL emission of ZnO indicates that the UV NBE is owing to the ZnO plane′s mainly from (002) preferred orientation [23]. ZnO takes crystal structure owing to the recombination of electrons and holes in the conduction and valence band, respectively. Mandal et al. [24] reported that the bandedge transition of ZnO film at 385 nm is mainly due to luminescence band. Depending on the microcrystalline structure and stoichiometry, the UV emission of ZnO film varies. Excellent crystalline structure obtained in our case was mainly because of NBE band. The absence of extra peak in the observed spectra explains the absence of intrinsic host lattice side effects that arises during film deposition. Moreover, the position and crystalline structure of thin films is dependent on proper sputtering power. When sputtering power was increased gradually from 10 W to 40 W, the peak intensity of UV emission also increased without any extra emission loss, and thereafter decreased when the sputtering power is further increased to 50 W. At lower sputtering power, the out diffusion of oxygen occurs resulting in oxygen vacancies. When sputtering power was increased from 10 to 40 W, intrinsic defects that arise due to zinc vacancy, oxygen vacancy, interstitial zinc, interstitial oxygen and antisite oxygen are decreased, thus giving an outcome of increased intensity and highly efficient NBE luminescence. When compared to results reported by Wang et al. [25], for ZnO film at high sputtering power and at room temperature using radio frequency magnetron sputtering, our samples show strong and defectless UV emissions, which is advantageous for optoelectronic device fabrication. At 50 W the oxygen vacancies tend to occur since zinc atoms decompose before reaching the substrate surface. The zinc ion bombardment may happen in ZnO target resulting in the degradation of ZnO film stoichiometry. Thus, at 50 W, due to intrinsic defects, the PL intensity of ZnO film decreased. Because of these observations, it is disclosed that the light emission characteristics of the ZnO film is greatly

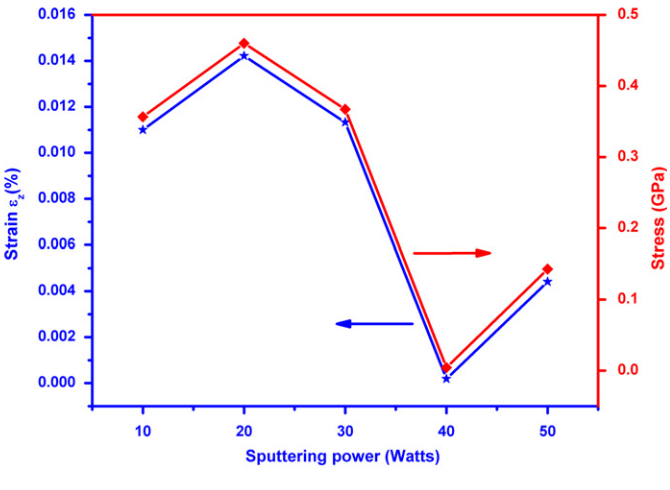

Fig. 4. The plot of strain and stress in ZnO films as a function of sputtering power.

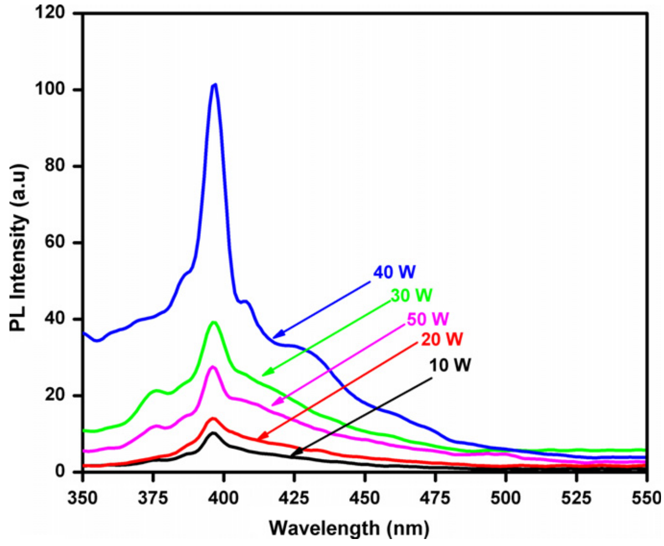

Fig. 5. Photoluminescence spectra of ZnO films prepared for different sputtering power.

influenced by the sputtering power, which in turn affects the orientation of the film, since orientation mainly depends on disorder or defects at the crystal planes.

3.3. *Effect of sputtering power on the optical properties of ZnO films*

In order to utilize ZnO for various applications, it is very important to understand the energy band structure of zinc oxide. Depending upon the band structure of the semiconductor materials, the electronic state of the excitons exists [26]. The excitons may be bound to neutral or charged donors or acceptors. The transmission and absorption processes are influenced by the dopant impurities or point defects created in the material. Interference of light occurs between the surface of the film and substrate material, which in turn affects the transmission spectra. The optical transmittance and absorption spectra of the ZnO film deposited on c-cut sapphire substrate at different dc sputtering power in the UV–visible wavelength range are shown in Figs. 6 and 7, respectively. From Fig. 6, it is inferred that in the electromagnetically visible spectrum range (600–900 nm), the ZnO film deposited at different sputtering power was highly transparent, and the average transmittance value reached upto 91%. The sharp absorption edges are seen in UV region, which is inferred from the commencement of fundamental absorption region seen in Fig. 7. The deposited ZnO film shows obvious improvement in the transmittance spectra. At 40 W, maximum transmittance was received due to removal of organic species in the film, and thus exceptional transparency was achieved when compared to other samples. At 50 W, the film transmittance decreased due to photon scattering and absorption in the film. This scattering was due to film surface morphology and point defects observed in the film. From Fig. 7, it is inferred that the absorption characteristics were greatly influenced by sputtering power. The absorption in visible region explains the localized energy states in the bandgap. In the UV region, at about 390 nm, exciton absorption edge was observed in the samples. Due to strain and defects in the film, the absorption edges slightly shift towards longer wavelengths. For the film deposited at 40 W the absorption edge was observed at the lower wavelength contributing to minimum strain. The optical bandgap 'E_g' of the ZnO film was determined by extrapolation of the linear portion of α^2 versus hv curve, using the following equation [27]:

$$(\alpha hv)^2 = A\left(hv - E_g\right)^n \tag{5}$$

where, A is absorption coefficient, hv is photon energy, E_g optical band gap energy and A is constant having values between 1×10^5 and $1\times10^6\,\mathrm{cm}^{-1}\ \mathrm{eV}^{-1}$. The measured optical band gap of ZnO films vary from 3.12 to 3.23 eV, which is very close to the band gap of bulk ZnO and are in good agreement with the PL results. The decrease and increase in band gap values were attributed to the increase and decrease in strain in the films.

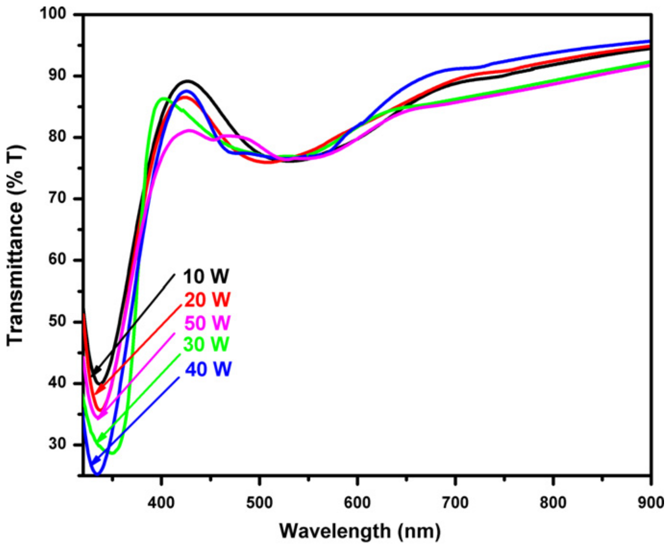

Fig. 6. UV transmittance spectra of ZnO films prepared for different sputtering power.

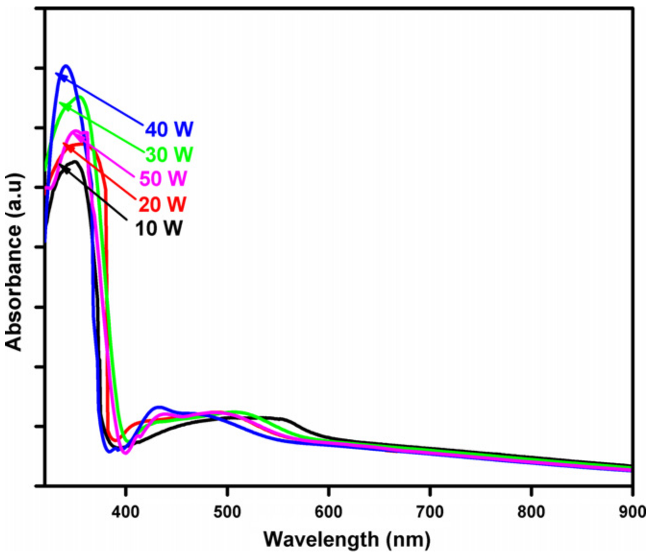

Fig. 7. UV absorbance spectra of ZnO films prepared for different sputtering power.

3.4. *Effect of sputtering power on the grain distribution and morphological properties of ZnO films*

Scanning electron microscope (SEM) produces high resolution images of the sample by scanning it with a focused beam of electrons [28]. In order to study the effect of sputtering power on the morphology of the films, SEM analysis was carried out for the films deposited at different sputtering power. Fig. 8 shows the SE-micrograph of ZnO films prepared at different sputtering power. The micrographs show that, the surface morphology of the films is significantly dependent on the sputtering power. The different dc sputtering power leads to different qualities of the crystalline lattice of ZnO films. The film prepared at

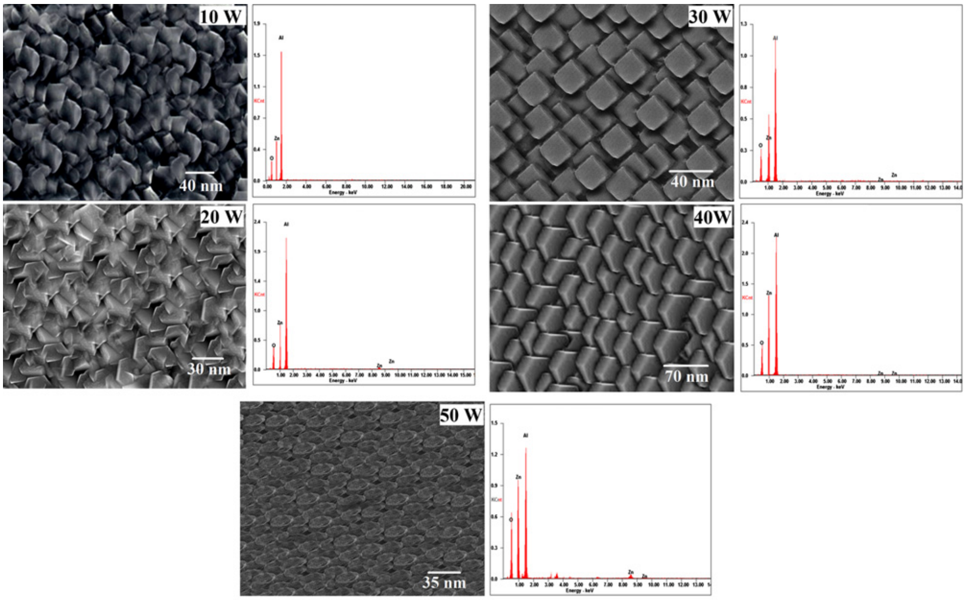

Fig. 8. HRSEM and EDS images of ZnO films prepared for different sputtering power.

10 and 20 W consists of nanoflake like grains with uniform coverage of substrate with randomly oriented grain distribution. At 30 W, the film revealed diamond shaped nanoparticulates with well defined corners. As can be seen from the images, the prepared films present good uniformity without any visible defects over the surface of the films, and the observation explains that the crystal quality can be continuously improved by increasing the dc power. The film prepared at 40 W shows that the morphology was changed completely to a block of hexagonal ZnO with more uniform grain distribution, indicating the increases in particle propagation through ion bombardment. Interestingly, increase in the sputtering power upto 40 W gives the deposited atoms extra surface mobility, allowing them to reach the favored position with very little mismatch on the surface of the c-cut sapphire substrate. This revealed a noticeable effect on the morphology of the film. The size of the grain observed in SEM image is in good agreement with the crystallite size calculated by Scherrer′s formula. To further confirm the compositions, EDS analysis of the samples were made, and are shown in Fig. 8. The EDS measurement indicated that in addition to Al only zinc and oxygen were detected in the sample. With the increase in dc power the composition ratio of zinc and oxygen approach the near stoichiometry of ZnO. The observed SE-micrographs of our samples grown at low temperature and low sputtering power are better than that reported by Bang et al. [29] for c-plane ZnO on c-cut sapphire, depicting the fact that the sputtering parameters has significant effect on the morphology of the films. From the observed micrographs of these samples, it is inferred that lower sputtering power of ~40 W should be used to obtain well grown grains with uniform distribution of hexagonal wurtzite ZnO.

4. Conclusion

Highly (002) oriented block of nanostructured ZnO were grown on sapphire substrate at low sputtering power. The XRD result revealed that the unique *c*-axis orientation of ZnO films can be obtained for all samples. Sputtering power and nature of the substrate mainly decided the structural characteristics such as crystal size, intrinsic stress and residual strain. According to PL measurements, strong UV emission was received owing to exclusive (002) orientation, which in turn reveals that the light emission characteristic of ZnO is significantly influenced by dc power. From SEM analysis, it is revealed that when sputtering power is varied, the uniform surface morphology is subject to change. At the sputtering power of 40 W, hexagonal block of wurtzite ZnO with uniform grain distribution was obtained, which indicates strong adhesion and very little mismatch with the substrate surface. The above factor leads to a conclusion that structural, optical and morphological properties are strongly influenced by the dc power and nature of the substrate.

Acknowledgments

This work was financially supported by the University Grants Commission (Major Research Project-File no. 40-441/2011), which is gratefully acknowledged. The authors thank SASTRA University for providing the experimental facilities.

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