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# Growth of Highly Oriented ZnO Thin Film by Pulsed Laser Deposition Technique

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***Abstract:*** *C-axis oriented ZnO thin films were grown on sapphire substrate using pulsed laser deposition technique. The structural and optical properties of the films were studied using XRD, SEM and photoluminescence (PL) spectroscopy techniques. Growth temperature and oxygen pressure are found to be very critical in obtaining a preferred c-axis oriented film with minimum point defects and low stress. PL studies reveal a decrease in the defect emission from films grown with increasing temperature and at higher growth pressure. The broad bluish green luminescence peak is likely to be due to the presence of native defects related to the zinc interstitials, oxygen vacancies and oxygen antisite defects at Zn sites in ZnO lattice. ZnO thin film having minimum defects (stress free and low concentration of defects) and highly c-axis orientation was obtained at 600°C and 5 Torr oxygen pressure.*

***Key Word:*** *pulsed laser deposition, thin film, stress, point defects, photoluminescence.*

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## I. Introduction

ZnO, a wide bandgap (3.3 eV) semiconductor, has potential application in various fields including optoelectronic devices [1], chemical and gas sensors [2], biosensors [3], piezoelectric devices [4,5], spintronics [6] etc. C-axis oriented ZnO thin films are preferred for these applications due to their superior properties as compared to other crystallographic orientations. Furthermore, the device performance is known to be highly sensitive to the presence of stress and native defects in the thin film [3-10]. The refractive index of the material is influenced significantly with the presence of stress, which causes an unacceptable degradation in the performance of optical waveguide effect. It is identified that the characteristics of photonic devices degrade with the presence of stress in the ZnO thin films [8]. Bagnall et al. highlighted the importance of growth of defect free ZnO thin films, showing only near band edge (NBE) emission and no deep level emission for the development of optical devices including UV luminescent devices [11]. Therefore, for the realization of an efficient photonic device, it is very important to deposit a high quality c-axis oriented ZnO thin film with minimum stress and low native defects showing minimum deep level emission in the photoluminescence (PL) spectra.

Reports are available in the literature on the growth of either stress free [9,10] or the native defect free ZnO thin films [12,13]. It is important to emphasize that not much attention has been given in the literature on the deposition of preferred oriented ZnO thin films having both minimal defect related to visible luminescence and residual stress characteristics simultaneously. Mostly synthesised ZnO thin films are either in the state of extensive stress or having large amount of native defects which leads to emission in the visible range. Therefore, efforts are continuing worldwide to deposit the ZnO thin film with minimum defects and stress [10-18]. Rashmi et al. have successfully optimized the deposition parameters for the growth of stress free ZnO films, however, the native defects related to oxygen and zinc were reported to be present in these thin films revealed by the presence of prominent visible emission in the PL spectra of the films [10]. Cui et al. studied the effect of annealing temperature as well as annealing atmosphere on the structural and optical properties of ZnO thin films grown. They were able to minimize the stress in the films; however, the films had a large amount of point defects as observed in the PL spectra [17]. Saron et al. optimized annealing temperature on film deposited with buffer layer of GaN on $Al_2O_3$ substrate using thermal evaporation technique, to achieve films with minimum stress of 0.069 GPa, however their films had some amount of defects [18]. On the other hand, Choppali et al. have deposited ZnO thin films free from native point defects exhibiting only NBE emission and absence of any visible emission, but the films are polycrystalline and studies on the presence of stress are not highlighted [13].

ZnO exhibits one characteristic emission peak in the ultraviolet region (3.3 eV) due to exciton recombination at room temperature [19,20]. ZnO thin films are known to have various defect states resulting in emission in the visible spectral range. The origin of visible emission centre in ZnO is attributed to different intrinsic defects, such as, Zn vacancies ($V_{Zn}$), Zn interstitials ($Z_{ni}$), oxygen vacancies ($V_O$), oxygen interstitials

($O_i$) and oxygen antisites ($O_{Zn}$) [21,22]. The presence of stress and native defects in ZnO thin films are known to be very sensitive to the deposition process and growth conditions. Several deposition techniques such as rf-sputtering [1,23], molecular beam epitaxy (MBE) [24], chemical vapour deposition (CVD) [25] and pulsed laser deposition (PLD) [21,22,26], sol gel [13,27] and spray pyrolysis [28] have been used for the deposition of ZnO thin films. Each technique has its own advantages and disadvantages. Amongst them, PLD is a widely exploited technique for the reliable reproduction of target stoichiometry in deposited films with fast turn-around time [21,22]. Hence, in the present study preferred c-axis oriented ZnO thin films are fabricated on sapphire substrates using PLD technique. Efforts have been made to optimize the deposition parameters to obtain highly c-axis oriented ZnO thin films exhibiting minimum stress alongwith reduced point defect concentration revealed by minimum visible emission in the PL spectra. Therefore, the deposited ZnO thin films are having potential applications for photonic device applications. The relationship between photoluminescence properties of ZnO thin films with oxygen pressure and substrate temperature is investigated.

## II. Material And Methods

ZnO thin films were deposited on (0001) $Al_2O_3$ substrate using pulsed laser deposition (PLD) technique based on Kr-F excimer laser ($\lambda$= 248 nm; frequency = 5.0 Hz). The films were deposited by ablating a dense ceramic target (25 mm diameter) of ZnO in 100% $O_2$ gas environment. The ceramic target was prepared using conventional solid state reaction procedure using ZnO powder (99.999% pure procured from Sigma Aldrich) and sintered at 1000°C in air for 4 hours. Prior to deposition the chamber was evacuated to a base pressure of $10^{-6}$ Torr. The depositions of ZnO thin films of 500 nm thickness were carried out at a fixed target-to-substrate distance of 3 cm under varying processing oxygen gas pressures (0.5 Torr to 10 Torr) and substrate temperatures (300°C to 600°C). The crystallographic orientation of the as-grown ZnO thin film was investigated by X-ray diffractometer (Bruker D8-Discover) using Cu-K$\alpha$ radiation and surface morphology was studied by a scanning electron microscope (Zeiss Ultra Plus). The thickness of the deposited thin films was measured using a surface profilometer (Dektak 150). Photoluminescence measurements were carried out at room temperature using He-Cd laser (325 nm) as the excitation source.

## III. Result

### Structural and Morphological Studies

The as-grown ZnO thin films were found to be transparent, smooth and strongly adherent to the sapphire substrate. XRD patterns of the samples deposited at 0.5 Torr oxygen pressure and different growth temperatures (300°C to 600°C) are shown in Figure 1.

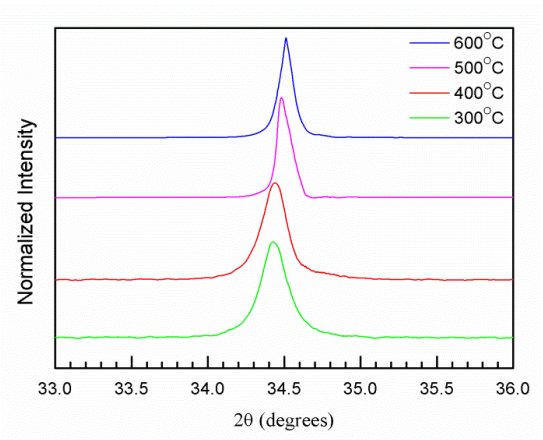


**Figure 1:** XRD spectra of ZnO thin films deposited at 0.5 Torr oxygen pressure and different substrate temperatures.

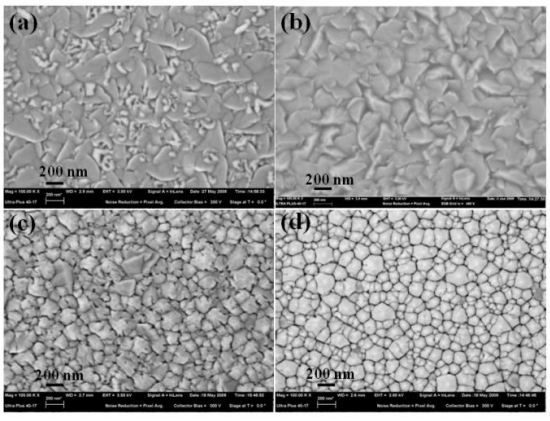


**Figure 2:** SEM image of ZnO thin films deposited at 0.5 Torr oxygen pressure and different substrate temperature of (a) 300°C (b) 400°C (c) 500°C (d) 600°C.

All deposited films were found to be (002) preferred orientation with c-axis normal to the substrate surface (Figure 1). Table no1 summarizes the values of various structural parameters estimated from XRD data including 2θ, lattice constant 'c', grain size and stress [23]. A continuous decrease in the full width at half maximum (FWHM) is observed for the ZnO thin films deposited with increasing growth temperature from 300°C to 600°C (Figure 1). This indicates that the grain size of the as-grown films increases (from 38 nm to 110 nm) with increase in growth temperature (Table no 1).

**Table no 1 :** The value of lattice parameter (c), grain size and stress of ZnO thin films deposited under varying growth parameters.

| Pressure (Torr) | Substrate Temp (℃) | 2θ Degrees | c (Å) | XRD Grain Size (nm) | Stress ($10^9$ N/m$^2$) |
|---|---|---|---|---|---|
| 0.5 | 300 | 34.42 | 5.206 | 38 | -0.07 |
| 0.5 | 400 | 34.44 | 5.204 | 41 | 0.18 |
| 0.5 | 500 | 34.47 | 5.199 | 87 | 0.56 |
| 0.5 | 600 | 34.51 | 5.194 | 110 | 1.07 |
| 0.8 | 600 | 34.50 | 5.195 | 95 | 0.94 |
| 1 | 600 | 34.48 | 5.197 | 89 | 0.69 |
| 3 | 600 | 34.46 | 5.200 | 95 | 0.43 |
| 5 | 600 | 34.45 | 5.202 | 97 | 0.31 |
| 10 | 600 | 3445 | 5.202 | 100 | 031 |
| Bulk | | | 5.206 | | |

It may be noted from Table no 1 that the estimated value of lattice constant 'c' for ZnO films deposited at low growth temperatures (< 500°C) is close to the corresponding value reported for bulk ZnO (5.206 Å). With increase in the growth temperature (≥ 500°C), a slight decrease in the value of lattice constant 'c' is observed indicating that the ZnO lattice is relatively compressed (~0.1% for the films grown at 500°C) along the crystallographic c-axis. The slight decrease in the value of lattice constant in comparison to that of bulk ZnO indicates the presence of stress in the ZnO thin films deposited at higher growth temperatures (≥ 500°C). The estimated values of the stress are also enumerated in Table no 1. These values are in the range (0.07 to 1.07)×$10^9$ N/m$^2$, which are much lower than the corresponding values reported by other workers on films grown by sputtering, pulsed laser deposition and sol-gel [8,29-32]. It may be noted from Table no 1 that the tensile stress (negative values) is present in films deposited at lower growth temperature (300°C) which becomes compressive (positive values) with the increase in growth temperature (≥ 400°C). The sample deposited at 600°C was found to possess a maximum stress of about 1.07×$10^9$ N/m$^2$. It is important to point out that the sample deposited at 300°C under an oxygen pressure of 0.5 Torr has minimal stress of about 0.07×$10^9$ N/m$^2$.

Figure 2 shows the SEM micro images of the ZnO thin films grown at a fixed oxygen pressure of 0.5 Torr and varying growth temperatures (300°C to 600°C). The growth of nanocrystallites occurs at lower temperatures (< 500°C). The surface morphologies of films deposited at higher temperatures of (≥ 500°C) are found to be well defined and uniformly distributed crystallites (Fig. 2). This is attributed to the increased mobility of the adatoms on the substrate surface at higher temperature that accelerates the migration of ablated species to the energetically favourable positions and help in the formation of good quality films with improved crystallinity. Well defined crystallites having hexagonal shape can be easily observed for films deposited at growth temperatures ≥500°C and these films are expected to have minimum defects.

ZnO thin films were also deposited at a fixed growth temperature (600°C) and varying oxygen growth pressure (0.5 to 10 Torr) and the corresponding XRD patterns are shown in Figure 3. Again, the XRD results indicate that all films are highly c-axis oriented. The estimated values of the lattice parameter 'c', grain size and stress are also included in Table no 1 for comparison. The values of 'c' are found to increase from 5.194 Å to 5.202 Å with increasing oxygen growth pressure from 0.5 Torr to 10 Torr. The value of 'c' (5.202 Å) obtained for the sample grown at 600°C and 5 Torr is close to the corresponding value (5.206 Å) reported for bulk ZnO [23], suggesting the absence of stress. The stress present in the films increases from 0.31×$10^9$ N/m$^2$ to 1.07×$10^9$ N/m$^2$ with decreasing oxygen gas pressure from 10 Torr to 0.5 Torr. However, the grain size of all samples deposited at different growth pressure is found to be almost constant (~95 nm). The film deposited at 300°C and 0.5 Torr oxygen pressure possesses minimum stress (0.07×$10^9$ N/m$^2$) followed by the film deposited at 600°C and 5 Torr oxygen pressure.

Figure 4 shows the SEM images of the films grown at fixed substrate temperature (600°C) and under varying oxygen pressure (0.5 Torr to 5 Torr). It may be noted from Fig. 4 that all samples show uniformly distributed hexagonal crystallites of approximately the same size (~100 nm) which is in agreement with the results obtained from XRD analysis (Table no1).

It may be inferred that the sample deposited at the temperature of 300°C and 0.5 Torr oxygen pressure has minimal stress whereas the film deposited at 600°C and 5 Torr pressure is highly preferred (002) orientation with negligible stress. The results indicate that the films deposited under optimized processing conditions have minimum non-radiative defects which generally originate due to the imperfection in crystallinity, grain boundaries and dislocations [29]. However, the possibility of radiative defects that originate from the presence of native point defects like oxygen vacancies, zinc interstitials, zinc vacancies, oxygen antisites etc. cannot be ruled out in the films.

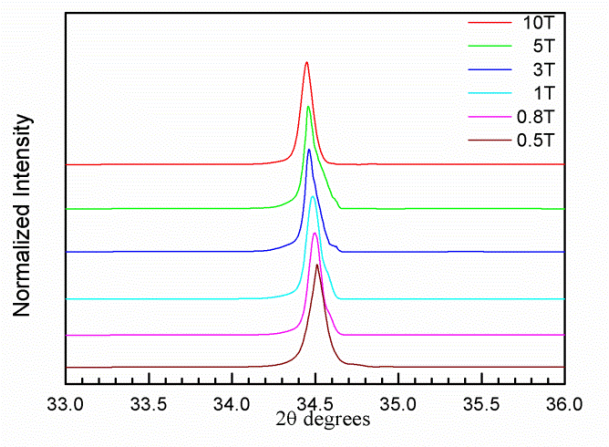


**Figure 3**: XRD spectra of ZnO thin films deposited at a fixed substrate temperature (600°C) and varying oxygen pressure

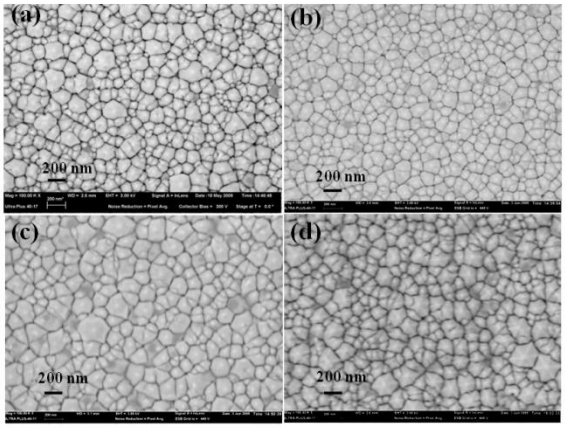


**Figure 4**: SEM images of ZnO thin films deposited at fixed substrate temperature (600°C) and varying oxygen pressure of (a) 0.5 T (b) 0.8 T (c) 3 T (d) 5 T.

**Photoluminescence (PL) studies**

The presence of possible native defects in the deposited ZnO thin films was studied using photoluminescence (PL) spectroscopy carried out at room temperature. Room temperature PL spectra of the samples deposited at a fixed pressure of 0.5 Torr and different growth temperatures (300°C to 600°C) are shown in Fig. 5.

Two broad emission peaks centered around 3.3 eV and 2.5 eV are observed in the PL spectra of all samples. The peak around 3.3 eV is the near band edge (NBE) emission and is the characteristic emission peak for ZnO originating from the recombination of free excitons [10,21]. The broad peak (~ 2.5 eV) observed in the visible region is the deep level emission due to the presence of point defects in ZnO films. It may be noted from figure 5 that for films grown at low growth temperatures (≤500°C), the NBE emission is weak in comparison to deep level emission indicating that these films have elevated levels of native point defects. The observed weak NBE emission may also be attributed to the presence of non-radiative defects [29] related to lattice imperfections in films grown at lower substrate temperatures ≤ 500°C as discussed earlier from SEM results. With the increase in growth temperature to 600°C, the intensity of NBE peak increases and this emission becomes more prominent as compared to the deep level emission suggesting that the point defects are minimal in ZnO thin films deposited at higher temperatures (600°C). It may be recalled that the SEM image of the film deposited at 600°C shows well defined and uniformly distributed hexagonal grains (Figure 2).

It is important to note from Fig. 5 that the NBE emission peak (3.3 eV) was sharp but asymmetric. The asymmetry in NBE peak may be attributed to the coupling of free excitons to longitudinal optical (LO) phonon replicas in polar ZnO thin films [19,20,33]. The results clearly indicate that the emission peaks are convolution of a number of peaks which may be related to different modes or presence of various defect states in the deposited ZnO thin films. Hence, each PL peak in Fig. 5 was deconvoluted using Gaussian approximations into three sub-peaks. The normalized intensities of various peaks obtained after deconvolution are listed in Table no 2 for all deposited ZnO thin films. The exciton-phonon coupling observed in the samples is due to the Fröhlich interaction and the deformation potential [34] and is generally observed in thin films and nanostructures, which is attributed to the presence of surface roughness [35]. The NBE peak was found to have the contribution from three sub-peaks centred at around 3.3 eV (376 nm), 3.24 eV (383 nm), and 3.17 eV (392 nm) and labelled as FE, FE-LO and FE-2LO, respectively. The FE peak corresponds to free exciton recombination whereas FE-LO and FE-2LO are LO phonon replicas [36]. The FE peak can be represented by the equation [19,33]:

$$FE(\lambda) = A \times \exp(-(\lambda-\lambda_{FE})/2\sigma^2) \quad (1)$$

where A is the Boltzmann factor, $\lambda_{FE}$ is the wavelength of the free exciton peak position and $\sigma$ is the variance. The energy difference between the free exciton recombination the FE-1LO and FE-2LO replicas is about 72 meV and 144 meV, respectively, and are in good agreement to values reported by other workers for bulk ZnO [36].

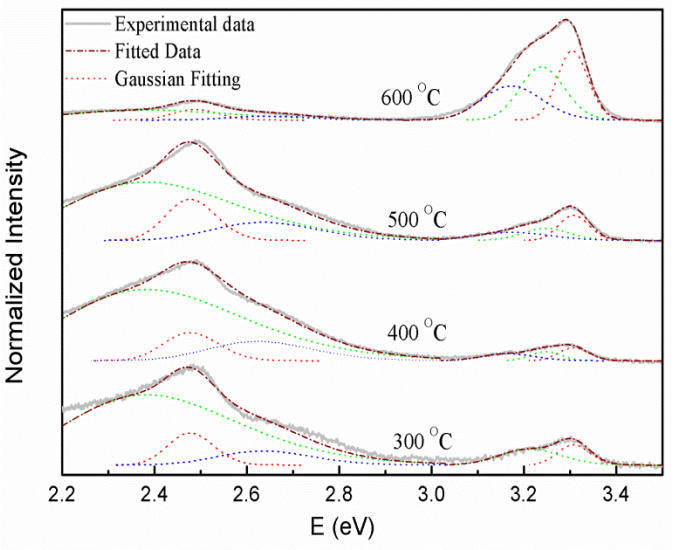


**Figure 5:** Room temperature PL spectra of ZnO thin films deposited at oxygen pressure of 0.5 Torr and varying substrate temperature.

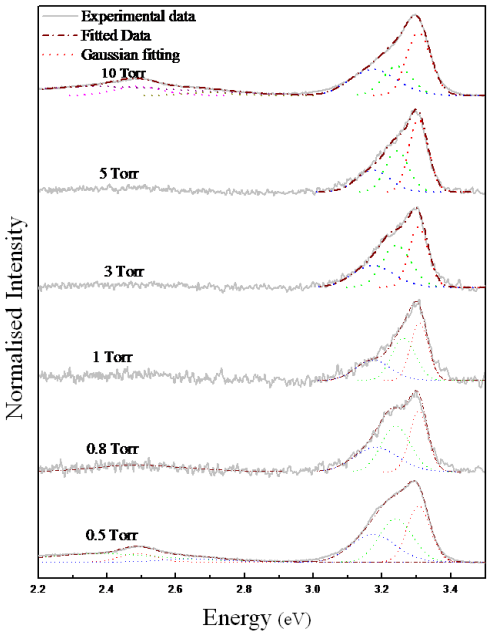


**Figure 6:** Room temperature PL spectra of ZnO films grown at substrate temperature of 600°C and varying oxygen pressure.

With an increase in the growth temperature from 300 to 600°C, the FE-1LO and FE-2LO emission peak intensities are found to increase (Table no 2), indicating its correlation with the increase in surface roughness as well as crystallinity of the layer. The increase in grain size with growth temperature results in a rough surface morphology thereby enhancing the contribution of exciton-phonon coupling and hence the intensities of the LO phonon replicas.

The deep level emission observed in the visible region in Figure 5, due to the presence of point defects in ZnO thin film is also deconvoluted into three emission peaks. The deconvoluted emission peaks at the energies of about 2.38 eV, 2.47 eV and 2.63 eV are due to oxygen antisites ($O_{Zn}$), oxygen vacancies ($V_O$) and zinc interstitial ($Z_{ni}$), respectively [21,37]. The intensity of defect emission peak with respect to the NBE peak is found to decrease with increasing growth temperature (Figure 5). Since the diffusion of atoms is small at lower substrate temperatures (≤ 500°C), Zn and O adatoms do not get enough time to settle onto their respective lattice sites. Therefore, the probability of formation of point defects in the ZnO thin film is higher in comparison to the one deposited at higher substrate temperatures (> 500°C), resulting in an increase of deep level emission at lower growth temperatures.

**Table no 2:** The intensities of de-convoluted emission peaks corresponding to NBE and deep level emission for ZnO thin film deposited under varying growth parameters.

| Pressure (Torr) | Substrate Temp.(℃) | FX (3.31 eV) | FX-1LO (3.24 eV) | FX-2LO (3.17 eV) | $Zn_i$ (2.63 eV) | $V_O$ (2.47 eV) | $O_{Zn}$ (2.38 eV) |
|---|---|---|---|---|---|---|---|
| 0.5 | 300 | 0.2 | - | 0.16 | 0.14 | 0.32 | 0.7 |
| 0.5 | 400 | 0.13 | 0.09 | 0.07 | 0.19 | 0.28 | 0.71 |
| 0.5 | 500 | 0.25 | 0.12 | 0.08 | 0.18 | 0.41 | 0.58 |
| 0.5 | 600 | 0.69 | 0.53 | 0.34 | 0.039 | 0.105 | 0.10 |
| 0.8 | 600 | 0.75 | 0.56 | 0.3 | - | 0.08 | - |
| 1.0 | 600 | 0.71 | 0.52 | 0.25 | - | - | - |
| 3.0 | 600 | 0.75 | 0.52 | 0.27 | - | - | - |
| 5.0 | 600 | 0.9 | 0.51 | 0.27 | - | - | - |
| 10.0 | 600 | 0.78 | 0.36 | 0.32 | 0.036 | 0.11 | 0.11 |

Room temperature PL spectra of the ZnO thin films deposited at 600°C (where the point defect concentration is minimal) and different oxygen pressures (0.5 to 10 Torr) are shown in Figure 6. The intensities and positions of all the deconvoluted peaks related to NBE and deep level emission are listed in Table no 2.

Figure 6 shows that all films deposited at 600°C have a well-defined and strong NBE emission peak irrespective of the growth pressure. However the intensity of the deep level emission is significantly reduced with the increase in oxygen pressure from 0.5 Torr to 5 Torr (Fig. 6) only to appear again at 5 Torr. The very low level of deep level emission peak for sample deposited between 1 – 5 Torr indicates minimal point defect concentration in these films.

It is interesting to note from Table no1 and Table no2 that the ZnO thin film deposited at low substrate temperature (400°C) and low oxygen pressure (0.5 Torr) are almost stress free. However, these films have poor crystallinity and large amount of point defects related to $Zn_i$, $V_O$, and $O_{Zn}$. On the other hand ZnO thin films deposited at higher substrate temperature (600°C) and moderate oxygen pressure (1 Torr) are almost free of point defects but are in compressive stress of about $0.69\times10^9$ N/m$^2$. Therefore careful optimization of the growth conditions is very important to obtain highly c-axis oriented ZnO thin films that have both minimal concentration of point defects and stress free condition. Films deposited at 600°C and 5.0 Torr oxygen pressure are almost free of point defects and have negligible stress ($0.31\times10^9$ N/m$^2$) that are suitable for device applications.

## IV. Conclusion

The effect of growth temperature and oxygen pressure on the presence of defects in preferred c-axis oriented ZnO thin films grown on sapphire substrate by PLD has been studied. The results show that growth conditions strongly affect the presence of stress and native point defect concentration in the films. PL spectra reveal intense NBE emission due to exciton-phonon coupling of the LO modes for films deposited at 600°C. Although ZnO films grown at lower substrate temperatures are stress free, they have large concentration of point defects. On the other side, ZnO thin films with minimal point defects are obtained at 600°C and 1 Torr but with the presence of significant stress in the films. The film deposited at 600°C and 5.0 Torr oxygen pressure has minimal point defects with negligible stress of $0.31\times10^9$ N/m$^2$. Therefore careful optimization of the growth conditions is very important for obtaining device quality ZnO thin films.

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