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The role of growth atmosphere on the structural and optical quality of defect free ZnO films for strong ultraviolet emission

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Abstract

Highly c-axis oriented wurtzite structured ZnO thin films were deposited on silicon substrates using pulsed laser deposition (PLD) by ablating a ZnO target in different atmospheres, including vacuum, argon and oxygen in the deposition chamber. The stress in the films was shown to vary from −3.83 to −0.03 GPa as a function of the chamber atmosphere. The minimum compressive stress (−0.03 GPa) was observed for the oxygen atmosphere. X-ray photoelectron spectroscopy data indicated that the O1s peak consists of three components designated as O1 (due to ZnO), O2 (due to defects) and O3 (due to adsorbed species). A small defect level emission was obtained in the luminescence spectra of the ZnO film deposited in the oxygen atmosphere, while strong ultraviolet (UV) emission was observed for the ZnO films deposited in the vacuum and argon atmosphere. These PLD grown ZnO thin films have the potential to be used as sources of UV radiation in light emitting devices.

Keywords: ZnO, PLD, XPS, NanoSAM, UV emission

(Some figures may appear in colour only in the online journal)

1. Introduction

In recent years, metal-oxide semiconductor films have been widely studied and have received considerable attention because of their properties and their applicability in several types of optoelectronic devices. In particular, the II-VI nanocrystalline compound semiconductors with tunable energy levels are promising building blocks for novel optoelectronic devices. Among the II-VI compound semiconductors, zinc oxide (ZnO) plays an important role in the preparation of optoelectronic devices [1] because of its excellent properties of piezoelectric, electron conductivity and a large exciton binding energy of 60 meV. Knowledge of the composition, optical, structural properties and impurity effects of ZnO nanocrystalline films is very important for such applications. The crystallinity and growth orientation play a very important role in the optoelectronic properties of the ZnO films. There are several deposition techniques used to grow ZnO thin films, including chemical vapour deposition (CVD) [5], magnetron sputtering [6], spray pyrolysis [7], the sol-gel method [8] and pulsed laser deposition (PLD) [9]. In the case of PLD films, the degree of orientation is influenced by the deposition conditions such as temperature, background gas composition and pressure, and kinetic energy of the plume particles [2]. Kinetic energy is mainly dependent upon the laser power density because the distribution of plasma and species can be varied by levels of laser fluence [3, 4]. In comparison with other techniques, PLD has many advantages such as stoichiometry transfer; high quality films at room temperature and ionized species in the laser-produced plasma [10]. However, the problem of efficient doping and controlling the properties of the films has remained to be solved as another technological issue. Various authors reported the synthesis of ZnO thin films by PLD for different optoelectronic applications [11–14]. However, synthesizing defect-free ZnO thin films is a big task for applications in many ultraviolet (UV) optoelectronic devices such as short wavelength light emitting diodes [15]. In this paper, the effect of different growth atmospheres on the luminance
properties of ZnO thin films is investigated. A correlation between stress and particle size (growth mechanism) has also been investigated. The role of growth atmosphere on the thickness as well as luminescence intensity is discussed in detail.

2. Experimental details

ZnO thin films were deposited on polished Si wafers using a 99.999\% pure ZnO target in a reactive PLD process. ZnO powder synthesized by solution-combustion method [16] was used for preparation of the target. The optimization and characterization of ZnO powder has already been reported elsewhere [16]. A 266 nm Nd:YAG laser was used for the ablation. The silicon substrate was cleaned ultrasonically using acetone, ethanol and deionized water; after that it was dried by N2 gas. The substrate temperature was kept at 300 °C during deposition of the thin films in a vacuum chamber. The laser energy and deposition time were fixed at 40 mJ and 25 min, respectively. The chamber was pumped down to a background pressure of 5 × 10−5 mbar and was then backfilled with O2 and Ar gases to a partial pressure of 5 × 10−2 mbar.

The structural properties were analyzed with an x-ray diffractometer (XRD) (PANalytical X’Pert Pro). The surface morphology and roughness of the films were examined by images captured in the contact mode using a Shimadzu SPM-9600 atomic force microscope (AFM). The root mean square (RMS) roughnesses were estimated by analyzing the topographic scans of the films. The x-ray photoelectron spectroscopy (XPS) analysis was carried out with a PHI 5000 Versaprobe-Scanning XPS Microprobe before and after 30 s of sputtering with Ar+. A PHI 700 Auger Nanoprobe was used for the depth profile analyses. A 25 keV, 10 nA electron beam was used. The Auger peak to peak heights (APPHs) were monitored while sputtering with 2 keV, 2 mA Ar+ ions using a raster that scanned the Ar+ ion beam over a 2 × 2 mm2 area, and the sputtering rate was 8.5 nm per minute. Photoluminescence (PL) data were recorded using a He-Cd laser with a 325 nm excitation wavelength. All characterizations were done at room temperature.

3. Result and discussion

3.1. XRD and structural analysis

Figure 1 shows the XRD patterns of the ZnO thin films deposited on Si substrates in three different background atmospheres. According to the XRD patterns, all the ZnO films are oriented along the (002) plane. This is in line with the characteristics of the hexagonal ZnO wurtzite where the c-axis is perpendicular to the substrate plane [17]. Fejimura et al [18] suggested that, in the ZnO crystal, the surface energy density of the (002) orientation is the lowest. Grains with the lowest surface energy become larger as the film grows and growth orientation develops at the crystallographic direction of the low surface energy. This implies that a (002) oriented film is formed in an effective equilibrium state that gives enough surface mobility to ad-atoms.

The crystallite sizes of the ZnO were calculated using the full width at half maxima (FWHM) of the (002) peak using Scherer’s formula given below [19]

\[ D = \frac{K\lambda}{\beta \cos \theta} \]  

where \( D \) is the size of the crystallites, \( \lambda \) is the wavelength of CuKα radiation (0.1514 Å), \( k \) is a correlation factor (0.94), \( \beta \) is the FWHM of the (002) peak and \( \theta \) is Bragg’s diffraction angle. The crystallite size of ZnO was 23 ± 1 nm. The numerical values of the lattice spacing for the ZnO films were also calculated, see table 1, from the XRD data according to the following equations [20]

\[ 2d_{hkl} \sin \theta = n\lambda \]  

where \( d_{hkl} \) is lattice spacing of \((hkl)\) and \(\theta\) is the Bragg angle (half of the peak position angle). For the wurtzite structure, the interplanar distance of the \((hkl)\) plane is related to the lattice parameters \(a\) and \(c\) via the Miller indices \(hkl\)

\[ \left( \frac{1}{d_{hkl}} \right)^2 = \frac{4}{3} \left( \frac{h^2 + k^2 + l^2}{a^2} \right) + \frac{l^2}{c^2} \]  

where \(a\) and \(c\) are the lattice constants; \(h, k, l\) are Miller indices. With the first order approximation \(n = 1\), the lattice constant \(a\) was calculated by [21]

\[ a = \frac{\lambda}{\sqrt{3} \sin \theta}. \]

For the (002) plane, the lattice constant \(c\) was calculated by

\[ c = \frac{\lambda}{\sin \theta}. \]  

The lattice constants \(a\) and \(c\) of bulk ZnO were determined as \(a = 3.2498 \text{ Å}\) and \(c = 5.2066 \text{ Å}\) [22]. The lattice structure of the substrate has an important impact on the crystalline structure and orientation of the film. The difference in the lattice structure between the substrate and the film also results in cracking, exfoliation or wrinkling of the film, and the dislocations at the interface between the substrate and the film can affect the crystalline quality of the thin film [23]. The similar lattice structure...
between the substrate and the film is beneficial to obtain high quality thin films. The lattice mismatch between the ZnO thin film and the Si substrate is 19%. As a result, the lattice constant \( d \) of the deposited ZnO films is shown in table 1. The value of the lattice parameter changed with a change in the background atmosphere. This may be due to the lattice contraction resulting from the presence of dangling bonds on the surface of the ZnO films. The ions on the surface of the ZnO films are incompletely coordinated and possess unpaired electrons. These dangling bonds (Zn\(^{2+}\) and O\(^{2-}\) ions) form an electric dipole, resulting in a parallel array of dipoles originating in the boundary layer of each particle, which lies on this surface and experiences a repulsive force. ZnO has the property of absorbing O\(^{2-}\) ions on the surface, which increases the electrostatic attractive interaction between Zn\(^{2+}\) and O\(^{2-}\) ions, resulting in lattice contraction \[22\]. The peak position as indicated for the (002) peak shifted to the right as the atmosphere was changed from vacuum to oxygen. The shifting in the peak positions is attributed to the change in the stress in the films \[24\]. There are two types of stress associated with thin films and these can mainly be ascribed to two reasons: one is the intrinsic stress due to impurities and defects in the crystal, and the other is extrinsic stress due to lattice mismatch, growth conditions and mismatch in the thermal expansion coefficient of the film and substrate. The stress in the films was calculated by using the biaxial strain model \[24\]

\[
\sigma = \frac{2C_{13} - C_{33}(C_{11} + C_{12})}{C_{13}} \left(\frac{c_0 - c}{c}\right)
\]

where \( C_{13}, C_{33}, C_{11}, C_{12} \) are elastic stiffness constants for ZnO. \( C_0 \) (0.52055 nm) is the strain-free lattice parameter measured from a ZnO powder sample. The values of the stiffness constants were \( C_{11} = 209.7 \text{ GPa}, C_{12} = 121.1 \text{ GPa}, C_{13} = 105.1 \text{ GPa}, C_{33} = 210.9 \text{ GPa} \) \[24\]. The variations of the stress values for the different film are shown in table 1. The stress values varied from \(-3.83\) to \(-0.03\) GPa when the chamber atmosphere was varied from vacuum to oxygen. It can be noted that the nature of the stress in ZnO films is compressive, but for the oxygen growth atmosphere the compressive stress is observed much less with respect to the other growth atmospheres. This indicates that the ZnO thin films are almost stress-free for the oxygen growth atmosphere.

### Table 1. Effect of different growth atmospheres on the different parameters of the ZnO films.

<table>
<thead>
<tr>
<th>Samples name/ atmosphere</th>
<th>Lattice constant ‘c’ (Å)</th>
<th>‘a’ (Å)</th>
<th>Stress (GPa)</th>
<th>Particle size (nm)</th>
<th>Surface roughness (nm)</th>
<th>Band gap (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnO/Vacuum</td>
<td>5.2494</td>
<td>3.2146</td>
<td>-3.83</td>
<td>43</td>
<td>1.5</td>
<td>3.77</td>
</tr>
<tr>
<td>ZnO/Argon</td>
<td>5.2227</td>
<td>3.1982</td>
<td>-1.50</td>
<td>57</td>
<td>3.6</td>
<td>3.78</td>
</tr>
<tr>
<td>ZnO/Oxygen</td>
<td>5.2094</td>
<td>3.1901</td>
<td>-0.03</td>
<td>59</td>
<td>3.4</td>
<td>3.81</td>
</tr>
</tbody>
</table>

The chemical analysis of ZnO thin films was performed by XPS. All binding energies were corrected for the charge shift using the C1s peak of graphitic carbon (BE = 284.6 eV) as in \[25\]. The detected carbon is related to the carbon adsorbed on the surface during the exposure of the samples to the atmosphere. The XPS spectra of the Zn 2p peak are shown in figure 2. The Zn 2p spectrum shows a doublet, whose binding energies are 1021.2 and 1044.3 eV, which can be ascribed to Zn-2p\(_{3/2}\) and Zn-2p\(_{1/2}\) doublets/lines. The binding energy difference between the two lines is 23.1 eV, which is comfortably lying close to the standard reference value of ZnO \[25\].

### 3.2. Chemical analysis by XPS

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The peak broadening was observed in case of the oxygen growth atmosphere samples before sputtering. This is due to some surface related defects. There is no broadening observed after 30 s Ar sputtering.

The deconvoluted curves of the oxygen O1s peak for ZnO thin films prepared in different growth atmospheres were...
The high-resolution spectra of the O1s peaks were measured before and after 30 s Ar⁺ ion sputtering. Kumar et al [27, 28] reported that the O1s peak of ZnO can be de-convoluted into three peaks, namely O1, O2 and O3. They attributed the O1 peak to O²⁻ ions on the wurtzite structure of a hexagonal Zn²⁺ ion array, surrounded by Zn atoms with their full complement of nearest neighbour O²⁻ ions [27–30]. The O2 peak is associated with O²⁻ ions that are in oxygen deficient regions within the matrix of the ZnO (such as ZnOH) [30] and the O2 peak represents the oxygen related defects in the system. It was impossible to fit the XPS peak of the unsputtered sample of the oxygen atmosphere sample with only two peaks (O1 and O3). A third peak (O2) therefore was added. This peak is in good

<table>
<thead>
<tr>
<th>Growth atmosphere</th>
<th>Peak position (eV)</th>
<th>Area (%)</th>
<th>Peak position (eV)</th>
<th>Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vacuum</td>
<td>O1 530.1</td>
<td>47.7</td>
<td>O1 530.2</td>
<td>89.8</td>
</tr>
<tr>
<td></td>
<td>O2 —</td>
<td>—</td>
<td></td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>O3 532.2</td>
<td>52.3</td>
<td>O3 532.6</td>
<td>10.2</td>
</tr>
<tr>
<td>Argon</td>
<td>O1 530.2</td>
<td>47.9</td>
<td>O1 530.2</td>
<td>84.5</td>
</tr>
<tr>
<td></td>
<td>O2 —</td>
<td>—</td>
<td></td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>O3 532.2</td>
<td>52.1</td>
<td>O3 532.3</td>
<td>15.5</td>
</tr>
<tr>
<td>Oxygen</td>
<td>O1 530.3</td>
<td>60.8</td>
<td>O1 530.2</td>
<td>86.4</td>
</tr>
<tr>
<td></td>
<td>O2 531.5</td>
<td>12.6</td>
<td>O2 —</td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>O3 532.4</td>
<td>26.5</td>
<td>O3 532.2</td>
<td>13.6</td>
</tr>
</tbody>
</table>

Figure 3. Deconvoluted peak of O1s into three peaks of ZnO in the different growth atmospheres before and after 30 s Ar⁺ sputtering.
agreement with the oxygen deficiency peaks found in previous work where defect emission was more pronounced. This peak is again therefore an indication of defects in the oxygen atmosphere thin film and it is also confirmed by the PL deep level emission (DLE) in figure 8(a) below. The O3 peak is usually attributed to chemisorbed species (such as CO3, adsorbed H2O or O2) on the surface of the ZnO [31]. The O2 peak was observed only in the oxygen atmosphere ZnO thin films and it vanished after sputtering. This indicates that the ZnO thin films synthesized in the oxygen environment have some oxygen related defects on the surface, which in turn affect the emission behaviour of the ZnO films. This kind of oxygen related defect on the surface was not observed in the films grown in the other atmospheres (vacuum and argon). The intensity of the O3 peak has decreased after sputter cleaning due to the removal of surface contaminants. The deconvoluted peak detail of the O1s peak is given in table 2.

3.3. Surface morphology and roughness analysis

The surface morphologies of the ZnO film synthesized in different growth atmospheres are shown as two and three dimensional AFM micrographs (500 × 500 nm²) in figure 4. The roughness of the ZnO thin films varied from 1.5 to 3.6 nm with the variation in the growth atmosphere. The particle size distributions were found to obey a log normal nature [32].

Figure 4. AFM images of ZnO thin films in different growth atmospheres.

Figure 5. Plot between the particle size and the stress of the film.
Here $d$ and $\sigma$ are related to the average size and the size distribution of the particles. By fitting the frequency plot using the above equation (solid blue line in figure 4), the average particle sizes of the ZnO at different atmospheres were determined and are given in table 1. The particle size was 43, 57 and 59 nm in diameter for films grown in vacuum, argon and oxygen atmosphere respectively. The particle size is directly correlated with the stress of thin films, observed by XRD results. The size of particles shows exponential growth with respect to the stress. The particle size is calculated with the given mathematical formula

\[
P(d) = \frac{1}{d \sigma \sqrt{2\pi}} \exp \left( -\frac{\ln^2 \left( \frac{d}{\sigma} \right)}{2\sigma^2} \right).
\]

where $a$ (59.5), $c$ (−4.8) and $k$ (1.01) is the constant. The graph of particle size as a function of stress is shown in figure 5.

3.4. Auger peak to peak heights (APPH) analysis

To examine the chemical composition of the ZnO thin films grown on the Si substrates in different atmospheres, Auger electron spectroscopy was used. The SEM and nano scanning Auger electron microprobe (NanoSAM) mapping images of ZnO films are presented in figure 6. The thin surface of the ZnO film deposited in the vacuum atmosphere

Figure 6. (a) SEM image of ZnO thin films deposited in different growth atmospheres. (b) Elemental mappings of ZnO thin films for different growth atmospheres: red, green and blue represent Zn, O and C, respectively.
is very smooth, while for the argon and oxygen atmosphere deposited films are apparently similar. The NanoSAM mapping of the films is also shown in figure 6. The elemental mapping of the ZnO thin film with the colors red, green, and blue, representing Zn, O, and C respectively, were performed in a 5 µm field of view to determine the elemental distribution of Zn and O elements from the ZnO film and Si from the substrate.

The depth profiles performed by NanoSAM for the ZnO films are shown in figure 7. The thickness of the films varied with growth atmosphere. The thickness of the films was 100, 60, and 175 nm for the vacuum, argon, and oxygen growth atmosphere, respectively. This spectroscopy, coupled with ion etching, has allowed us to evaluate the film composition throughout its thickness to examine evidence of compositional variation during film deposition. In all the depth profiles, the main elements present in ZnO, i.e., zinc (Zn) and oxygen (O), were detected. In addition, the adventitious carbon (C) on the surface and silicon (Si) from the substrate were also detected. The atomic concentrations of Zn and O were found to be almost constant through the thickness of the samples, which clearly indicates a homogeneous composition of the film throughout its depth; but a variation of the adventitious C on the surface was observed. The C on the surface was immediately removed when sputtering was started for the argon and oxygen sample.

3.5. Growth mechanism

In general, the interaction between the laser radiation and the solid material takes place through the absorption of photons by electrons of the atomic system. The absorbed energy causes electrons to be in excited states with high energy, and as a result the material heats up to very high temperatures in a very short time. Then, the electron subsystem will transfer the energy to the lattice by means of electron–phonon coupling [33, 34]. If the laser energy is sufficient for ablation of the source target, the material evaporates, forming a gas plasma with the characteristic shape of a plume. This plasma plume expands along the direction normal to the target surface. When it reaches a substrate placed in front of the target, part of the evaporated material will form a thin film on this substrate. In particular, the plasma expansion dynamics, which plays a crucial role in pulsed laser deposition, is not yet totally understood, even in the simpler case of propagation in a vacuum. Furthermore, the expansion behavior is even more complex in the presence of an environmental gas, often used during PLD deposition of complex oxides. In such a case, additional physical processes affect the plume expansion behavior, such as
plume deceleration and splitting, shock-wave formation, thermalization etc, as a consequence of plasma–background gas interaction [35]. The deposition process occurs in a vacuum chamber to minimize the scattering of the particles. In some cases, however, reactive gases are used to vary the stoichiometry of the deposited film [35]. In case of a vacuum, the plume angular distribution is determined by the collisions of the plume particles among themselves in the initial stage. When the plume is small however, in the presence of the atmosphere gas, the plume angular distribution is modified due to collisions between the plume species and background gas atoms [36]. These collisions scatter the plume particles in the form of ZnO clusters, Zn^{+} ions and Zn^{2+} ions, and these plumes are affected by the growth atmosphere. Three cases in film growth may be possible in the work, reported below.

Case I. In a vacuum, re-sputtering of ZnO from the film surface is also possible because there is no partial pressure of gas inside the chamber. A much smoother layer was formed with fewer defects in the layer.

Case II. In an argon growth atmosphere, only ZnO clusters are deposited on the substrate. Zn^{2+} ions are not going on the substrate such that the thickness in the argon atmosphere is less than the vacuum.

Case III. The Zn^{2+} ions reacted with O^{2−} ions provided by the oxygen atmosphere and the number of ZnO clusters is increased in this case resulting in the maximum thickness for films grown in the oxygen atmosphere. The probabilities of ZnO formation during the plasma expansion through one of the following reactions

$$\text{Zn}^+ + \text{O}_2 \rightarrow \text{ZnO}^+ + \text{O}$$

$$\text{Zn}^2+ + \text{O}_2 \rightarrow \text{ZnO} + \text{O}$$

leaving some extra oxygen that forms part of the oxygen deficiency regions.

3.6. Photoluminescence study

The PL spectra of ZnO films grown in different atmospheres are shown in figure 8(a). It is worth noting that the films mainly exhibit emission in the UV region. The strong near-band edge emission at room temperature is due to free exciton recombination. In undoped ZnO, the visible light emission is ascribed to the structural defects [37] such as zinc vacancy ($V_{\text{zn}}$), oxygen vacancy ($V_{\text{o}}$), interstitial zinc ($Z_{\text{zn}}$), interstitial oxygen
(O_i) and antisite oxygen (O_{zn}) [38–40]. Disappearance of visible emission implies that the ZnO films have very low defect density and excellent optical quality. The intensities of different emissions in ZnO result from the competition between the different recombination mechanisms. UV emission originates from the free exciton recombination, and broadband visible emission is ascribed to the recombination related defect levels. In the as-grown nanocrystalline ZnO films, the absence of visible emission gives rise to the strong UV emission. The full width at half maximum (FWHM) of PL for ZnO films is 153 meV, larger than the 80 meV of single-crystal ZnO. The cause of broadening could be homogeneous and inhomogeneous strain related to lattice parameter and thermal expansion coefficient mismatches between substrate and film [41]. The peak position of band to band emission (BBE) is shifted from 376.7 to 380.5 nm when the growth atmosphere was varied from vacuum to oxygen. The change in the peak position is directly correlated with the stress of the thin films. The variation of stress with peak position is shown in figure 8(b) and is dependent on the equation given below.

\[ \lambda = A_1 + \frac{(A_2 - A_1)}{1 + 10^{[\log_{10}(x)]}}. \]

Figure 8(c) shows the schematic band diagram of the emissions in the ZnO constructed from the data. The position of the theoretical value of the Zn_i level is at 0.22 eV below the conduction band [42]. The green emission from ZnO is explained by the full potential linear muffin-tin orbital method, which suggests that the position of the O_i level is at 2.28 eV below the conduction band [43].

4. Conclusion

C-axis oriented ZnO thin films were deposited successfully on (100) p-type Si substrates in different atmospheres by the PLD technique. The XPS results of the O1s peak and the PL data confirmed that there were fewer defects in the ZnO structure. A well-known UV emission was observed from the ZnO films with the films deposited in vacuum and oxygen giving respectively the least and highest PL intensity. The particle size and emission intensity were dependent on the growth atmosphere and stress. These ZnO thin films can be used as a suitable future material for UV laser applications.

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