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The effect of Oxygen Mixing Percentage on structural, optical and electrical properties of ZnTiO₃ thin-films grown by RF magnetron sputtering.

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Abstract:

Perovskites are important composites in the area of multidisciplinary applications. It is achieved by carefully choosing and tuning the properties of the thin-film at the deposition. In this paper, ZnTiO₃ (ZTO) thin-films were being deposited on quartz and N-Si substrates by RF magnetron sputtering. The thin-films were developed at room temperature, oxygen percentage levels varying from 0 to 100, and annealed at 600°C. The electrical, optical, morphological, and structural properties were analyzed as a function of oxygen mixing percentage (OMP). The crystallinity of the cubic structured ZTO thin-film is found to be high at 25 OMP, and it is gradually decreased with increased OMP. The surface morphology of the thin-film is observed, and roughness is measured from the atomic force microscope. Raman Spectroscopy investigated the phase formation and the vibrational modes of the thin-film with their spectral de-convolution. The ZTO thin-films optical properties were investigated using transmittance spectra. The ZTO thin-film indicated the highest refractive index of 2.46, at 633nm with optical bandgap values of 3.57 eV, with a thickness of 145nm and 25 OMP. The refractive index, thin-film thickness, and excitation coefficient were analyzed using the Swanepoel envelope technique. Electrical characteristics of ZTO thin-film are measured from the optimized conditions of the thin-film with conventional thermionic emission (TE) technique.

Keywords: Thin-film; Optical properties; Swanepoel envelope technique.

1. Introduction:

For several decades, metal oxide semiconductor (MOS) thin-films play a significant role in the interdisciplinary research areas, and its applications. In the present scenario, researchers are working on MOS composites. Generally, metal oxide composites form ABX_3 perovskites. Here 'X' is anions such as O, F, I, N, or halogens. 'B' is transition metal elements such as Ti, Mg, Pb, Fe, Cu, Ta, Zr, Al, Cr, Mn. 'A' is metal cations such as Ca, Zn, Ag, Cs, K, Na, Cd, Pb, Ba, La[1]. Fuel cells, non-linear optics, memory devices, gas sensors, photocatalysis, solar energy conversion, water splitting, decomposition are some of the essential perovskite applications [2][3][4]. The metal oxides such as ZnO, TiO₂, SnO₂, MnO₂, CuO, WO₃ are known as wideband semiconductors [5]. Among these metal oxides, ZnO and TiO₂ thin-films have extensive applications of their intrinsic properties, mixed oxides formations, and transition metal doping. Incorporation of ZnO with TiO₂ leads to Zn-Ti-O ternary oxides which ensure the separation of electron-hole pair efficiently [6]. Under the light illumination, it contributes to optoelectronic devices' applications such as photovoltaic and dye-sensitized solar cells, light-emitting diodes, sensors, flat panel displays, and photodetectors. ZnO – TiO₂ composite exists in zinc Orthotitanate (Zn₂TiO₄, cubic spinel crystal structure), zinc titanate (ZnTiO₃, either cubic spinel or hexagonal perovskite structure), and zinc poly titanate (Zn₂Ti₃O₈, cubic spinel structure) [7][8]. The ideal cubic crystal structure of ZnTiO₃ is rare and often distorted with reduced symmetry[9].

However, To obtain good quality stoichiometric and crystalline thin-films: dopants, chemical composition, substrate temperatures, annealing temperatures, minimum thickness, required phase, and suitable oxygen atmosphere are very important. A small weight percentage (1 wt. %) of SnO₂ has doped to ZTO stoichiometric composition. It improves the properties of ZTO thin-film because a small quantity of SnO₂ can enhance the intensity of the cubic nature of ZTO thin-film and increases the surface adatom mobility of the charges, which leads to the coalition of smaller grains[10][11][12]. Several researchers studied the effect of oxygen mean pressures and concluded the impact of OMP on the thin-films structural, electrical, and optical properties. The phase composition, crystal structure, and optical behavior of the metal oxide thin-films can be controlled by adjusting the oxygen flow rate in the sputtering process [13- 16].

In this paper, a systematic study on structural, optical, morphological, and electrical properties of ZnTiO₃ thin-films deposited at oxygen mean pressures (OMP) variant by Radio Frequency sputtering is

reported. A small concentration of SnO_2 was doped to the ZTO sputtering target. The crystallinity and structure are determined with XRD. The thin-film optical constants, thickness, and excitation coefficients of ZTO thin-films were measured using the Swanepoel envelope technique. Roughness and morphology were observed from AFM. The best and optimized conditions were used to determine I – V characteristics of ZTO thin-film deposited on N-Si substrate with Ag electrodes. As per the author's knowledge, there are very few literatures available for I – V characteristics of ZTO thin-film.

2. Experimental Procedure:

The ZnTiO_3 (ZTO) thin-films were deposited on quartz and N-Si substrates using the RF magnetron sputtering method. The stoichiometric ZnTiO_3 (ZTO) with a small weight percentage (1 wt. %) of SnO_2 produced a sputtering target using the conventional solid-state reaction method. Zinc Oxide (ZnO , ~30nm, 99.8% pure, SSA ~35 m^2/g , Zincite phase), Titanium Dioxide (TiO_2 , ~35nm, 99.9% pure, SSA ~60 $\pm$ 20 m^2/g , Anatase phase), Tin Oxide (SnO_2 , ~80 nm, 99.9% pure, SSA ~ 11 m^2/g , cassiterite phase) elementary powders were mixed using a planetary ball mill (Fritsch GmbH, Germany) with prescribed stoichiometry. The mixed powders were dried to room temperature. The powders were uni-axially pressed to produce a ZnTiO_3 target of 60 mm diameter and sintered at 450 °C for 4 hours. Before deposition, the sputter chamber has been evacuated up to 1.0×10^{-6} m bar base pressure. To obtain 3.0×10^{-2} m bar pressure, the chamber is pumped with argon (Ar) and oxygen (O) gases. The sputtering power is fixed to 50W. The ZTO thin-films were deposited at room temperature and annealed at 600°C. For the uniform deposition rate, and the same thickness, different levels of oxygen (O) and Argon (Ar) gases were applied to the chamber. The thickness of the films and deposition rate were optimized using UV-VIS Spectroscopy (Veeco-Dektak 6M). The purity of phase and crystal structure of the thin-films were obtained using an X-ray diffractometer (Rigaku, TTRAX III 18 kW) with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Atomic force microscope (Agilent, 5500 series) is employed for the thin-films roughness and surface morphology analysis. ZTO (N-Si, Quartz) thin-film was fabricated, and a top electrode (Ag) was deposited by thermal evaporation. UV–VIS–NIR Spectrophotometer (UV 3101PC, SHIMADZU) is employed for the spectral transmission characteristics in the 200 – 2500 nm wavelength range. To study the vibrational modes and FWHM of the thin-film reviewed from Raman spectroscopy (LABRAM HR800, JOBIN YVON). I-V characteristics of the thin-film were obtained from a 4-point probe station and Keithley (4200 SCS). Table I represents the sputtering conditions of the ZTO thin-film.

3. Results & Discussions:

3.1 XRD:

Fig. 1(a) shows the X-ray diffraction (XRD) patterns of the ZnTiO₃ (ZTO) thin-films grown at room temperature under various oxygen atmosphere conditions (0-100%). It is seen that the thin-film deposited at room temperature is purely amorphous, which is indicating that no crystallization occurred. At 600°C, the ZnTiO₃ phase was found, and all peaks are confined to a cubic perovskite structure (ICDD:00-039-0190). No secondary phases were found in the composite [17][18].

Main reflections are obtained at (2 2 0), (3 3 1), (4 2 2), (5 1 1), (4 4 0) planes. The plane (3 1 1) at $2\theta = 35.58^\circ$ is the predominant peak. The highest intensity of peak at $2\theta = 51.75^\circ$ is Si substrate. Which indicates the orientation of ZnTiO₃ thin-film grown on N-type Si substrate. Crystallite size (P) is calculated from Scherrer approximation, which is defined as

$$P = \frac{0.9 \lambda}{\beta_{hkl} \cos \theta} \quad (1)$$

Where P is the crystallite size in nm, β_{hkl} is a full-width half-maximum of the peak in radians, λ is the wavelength of radiation (1.54056 Å for CuK α), and θ is Bragg angle.

The average crystallite size calculated with the equation (1) was increased from 3.5 nm to 6.19 nm, with an increase of OMP from 0 to 25. Crystallite size decreases from 6.19 to 4.2 nm with an increase of OMP from 25 to 100, which is confined to the nanocrystalline nature of ZnTiO₃. It can be correlated that, the sputtered atoms react with oxygen molecules which generates redistribution of energy and heat on the substrate's surface. This process concurrently promotes sputtered species migration and crystallization. For ZTO thin-films initially, O₂ helps the crystalline growth up to 25 OMP, then the growth gradually decayed up to 100 OMP. The trends in lattice volume, D spacing, crystallite size, lattice strain, and lattice constant with respect to OMP were calculated and represented in Fig.1(b). The thin film deposited at 12.5 % OMP to 25 % OMP is the better condition for ZnTiO₃ thin film fabrication.

3.2 AFM:

Fig. 2 shows the surface morphology, holographic roughness(internal), and typical 3-D representation of ZTO thin-films deposited with different OMP's. The images are obtained from Atomic force microscopy. The average roughness to be an increase from 0.25 nm to 0.74 nm with the increase of OMP from 0 to 25 and roughness decreases from 0.74 nm to 0.48 nm with the rise of OMP from 25 to 100. AFM micrographs follow the same trend observed from XRD. But the films deposited in 100% oxygen and 0% oxygen atmosphere exhibited a uniform and homogeneous but low dense microstructure with regards to surface topology and thickness. The grain growth enhancement may be optimized at 25 OMP for ZTO thin-film to improve the films' crystallization in oxygen and argon mixed atmosphere. The roughness value is < 1nm is depicted in ultra-fine thin-films deposited by the sputtering technique. The small roughness peaks can act as nanostructured absorption sites for sensing applications [19][20]. Fig.3 represents the RMS roughness of the deposited thin-films.

3.3 Optical properties (UV-Visible Spectroscopy) :

Fig. 4(a) depicts the transmittance spectra of ZTO thin-films fabricated on the quartz substrates. It is computed on a scale of 200-2500 nm wavelength. In the visible range, thin-films were transparent (>75%), and Fabry- Prot interference behavior was perceived. Because of the thin-films fundamental absorption, the transmittance was decreased to zero in the wavelength range of 235-265 nm. The absorption edges show a bathochromic shift with increasing the OMP. The optical constants were determined by using the Swanepoel envelope technique [21][22]. The refractive index computed from the following equation,

$$n = \sqrt{\frac{1}{2} \left(\sqrt{1 + \frac{4n_s^2}{(T_{\max} - T_{\min})^2}} + 1 \right)} \quad (2)$$

Where,

$$N = 2n_s^2 \left(\frac{T_{\max} - T_{\min}}{T_{\max} * T_{\min}} \right) + \frac{n_s^2 + 1}{2} \quad (3)$$

$T_{\max}$ is the transmittance maxima and $T_{\min}$ is the transmittance minima at a specific wavelength λ , and n_s is the substrate's refractive index. The thin-films thickness (d) can be calculated by the following equation,

$$d = \frac{\lambda_1 * \lambda_2}{2(n_1^2 * n_2 - \lambda_2 * n_1)} \quad (4)$$

n_1 and n_2 are the refractive indices of two adjacent maxima or minima at wavelengths λ_1 and λ_2 .

The films' thicknesses were in the range of 160-177 nm is almost constant. It is calculated that the refractive index (n) of the thin-films was from 2.35 to 2.46. It follows the same trend as XRD and AFM. usually, the refractive index depends on the thin-films crystallinity, electronic structure, and oxygen deficiencies.

The optical band gap energy (E_g) of the thin-films are acquired from the extrapolated linear portion of $(\alpha h\nu)^m$ vs $(h\nu)$ curve, where $h\nu$ is the photon energy, α is the absorption coefficient. The measure of crystalline order β related to the bandgap energy is $(\alpha h\nu)^m = \beta (h\nu - E_g)$. The bandgap energy (E_g) is calculated by considering an allowed direct ($m=2$) transition of electron between the highest occupied state of the valence band and the lowest unoccupied state of the conduction band. The thin-films absorption edges at different OMP were shown in Fig. 4(b). It is observed that the optical bandgap energy values are in the range of 3.39 – 3.60 eV. The bandgap variations might be due to reduced oxygen vacancies, variations in crystallinity, and improved grain size [23]. Fig. 4(c) represents the refractive index (n_f), absorption coefficient (α), Excitation coefficient (K), and optical energy bandgap (E_g) of the ZTO films concerning different OMP's.

3.4 EDS:

The Energy dispersive spectrum technique confirmed the elemental distribution of the ZTO composite. Fig 5(a) shows the typical microstructure, elemental mapping, and Fig 5 (b) depicts the energy-dispersive spectra of ZTO composite deposited on the N-si substrate at 25 OMP condition. The experimental volume fraction of ZTO composition is in agreement with theoretical volume fractions, confirming the ZTO composite's stoichiometry. The peaks in the spectrum are similar to X-ray diffraction peaks. The predominant peak in the spectrum is N- Si substrate, which represents the orientation of the thin-film on(1 0 0) N- Si substrate.

3.5 Raman Spectra:

Fig. 6 exhibits the Raman spectra of ZTO composite recorded in the wavenumber range from 50 to 1000 cm^{-1} and their spectral de-convolution, the spectrum was fitted with Gaussian function using origin pro software. Seven to eight active Raman modes were identified for deposited ZTO thin-film at different OMP. Table II represents the Raman modes and full width at half maxima. The bands persist cubic phase of ZTO, displays two large and broad bands at 310.464 cm^{-1} , 432.600 cm^{-1} , and all the spectral features are of the first order. The result can be explained based on the order-disorder model of the central Ti ion. According to group theory ZnTiO_3 has ten Raman active modes $5 A_g + 5 E_g$. The bands at 102 cm^{-1} , 153 cm^{-1} , 433 cm^{-1} , 583 cm^{-1} are the E_g modes and 310 cm^{-1} , 489 cm^{-1} , 795 cm^{-1} are related to A_g modes of the ZTO Raman spectra [24][25].

3.6 I-V Characteristics:

Fig. 7 shows the I – V characteristics of ZnTiO₃ thin-film on N-type Silicon substrate which deposited at 25 oxygen mixing percentage. The Ag electrodes were deposited on a thin-film with thermal evaporation sputtering unit. From the characteristics, it was observed that the thin-film was having a non-linear and symmetrical response for both forward and reverse bias. The high resistivity of the film is observed. In general, chemically deposited films have high resistance. It is due to low donor defect density and a large number of chemisorbed oxygen species. A large number of oxygen molecules are chemisorbed at the grain boundaries and on the thin-films surface. The surface resistance of the thin-film measured from the four-probe station is $R_s = 5.59 \times 10^9 \Omega$

Chemisorption is the process of trapping conduction electrons from the negatively charged oxygen species (O^{2-} , O^-). Due to this chemisorbed species, the resistivity of the oxide surface is high. For reducing gas molecules, the negatively charged chemisorbed species act as reaction centers. When reduction gases come in contact with the oxide surface, the trapped electrons releases because of the reaction between gas molecules and the oxygen species. The electrons return to the conduction band so the resistance is decreased. After the removal of the gas, the electrons are again trapped, and the resistance increases. Thus such high resistive films containing an enhanced density of chemisorbed species are particularly suitable for resistive mode gas sensor applications[26].

From the conventional thermionic emission (TE) theory, the forward I–V characteristics of the ZTO thin-film deposited on N-type Si substrate with Ag contacts can be delineated by

$$I = I_0 \exp\left(\frac{(V-IR_s)q}{\eta kT}\right) \quad (5)$$

Where I_0 is the reverse saturation current, V is the applied voltage, R_s is the series resistance, η is the ideality factor, q is the charge, k is the Boltzmann constant, and T is the absolute temperature.

The reverse saturation current I_0 is given by

$$I_0 = AA^*T^2 \exp\left(\frac{-q\phi_{B,eff}}{kT}\right) \quad (6)$$

where A is the contact area of ZTO ($\sim 0.765 \times 10^{-2} \text{ cm}^2$), A^* is the effective Richardson constant of ZTO ($\sim 37 \text{ A cm}^{-2} \text{ K}^{-2}$), and $\phi_{B, eff}$ is the effective barrier height at zero bias. It is described as

$$\phi_{B,eff} = -\frac{kT}{q} \ln\left(\frac{I_0}{AA^*T^2}\right) \quad (7)$$

Taking natural logarithm on both sides of Eq. (1), we obtain

$$\ln(I) = \ln(I_0) + \frac{q(V-IR_s)}{\eta kT} \quad (8)$$

For a low current region of the forward bias I–V characteristics, the forward bias current is in the order of I_0 , the effect of R_s is negligible due to the negligible value of IR_s . Thus, the reverse saturation current value I_0 can be calculated from the intercept of $\ln I$ versus V plot (shown in Fig. 8(a)) with the current(I) axis for $V = 0$, which gives $I_0 \sim 5.833 \times 10^{-11}$ A. The value of I_0 is then used in Eq. (7) to determine the value of $\phi_{B,eff}$ as 0.87 eV. This value may be deviated from the ideal value because of high surface states, generation-recombination, image force lowering effect in the depletion region, and the barrier inhomogeneities at the junction. Now, the ideality factor (η) is computed from the slope of the linear region of the forward bias $\ln I$ versus V plot as

$$\eta = \frac{q}{kT} \left(\frac{dV}{d \ln(I)} \right) \quad (9)$$

From Eq. (9), the ideality factor(η) is estimated as ~ 2.35 , which is much larger than unity. The high η values represent the interfacial thin oxide layer, a wide distribution of barrier height, and the bias voltage dependence of the barrier height[27][28].

To determine the value of the device's series resistance, we have used the $R_i = dV/dI$ vs V plot of the measured I–V data, as shown in Fig. 8(b). where R_i is the bias-dependent resistance. The series resistance is almost negligible at lower values of current. At higher values of current R_s shows a significant effect so that it exhibits nonlinear characteristics. In the high current region, the voltage drop IR_s is much larger than the voltage appearing across the ZTO thin-film and hence applied bias $V = IR_s$. The input resistance $R_i \sim 2.3 \times 10^9$ is determined from Fig. 8(c).

Conclusions:

RF magnetron sputtering was used to deposit ZTO thin-films on quartz, N-Si substrates. The influences of the OMP on structural, optical, morphological, and electrical properties were studied systematically. The deposited thin-films annealed at 600°C were crystallized in pure ZTO cubic phase without any secondary phases. The roughness value is < 1 nm is depicted in ultra-fine thin-films deposited by the sputtering technique. The thin-films refractive index is high at 25 OMP is 2.46, whereas the optical band gap varies from 3.4 eV to 3.6 eV with varying OMP. The stoichiometry of the thin-film meets the elemental composition. The vibrational modes of the Raman spectra representing the cubic structure of ZTO. The electrical parameters of the thin-films are Reverse saturation current I_0 is 5.83×10^{-11} A, Barrier efficiency is $\phi_{B, eff}$ is 0.87 eV, and ideality factor η is 2.35. The thin-film parameters are more suitable for optoelectronic, microwave dielectric, and gas sensing applications.

Acknowledgments:

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Figures

Figure 1

(a). XRD patterns of ZnTiO₃ thin-film at different Oxygen Mean Pressure (OMP) (Ar = 100 % to 0 %, O₂ = 0 % to 100 %). (b). Lattice volume, d spacing, Crystallite size, lattice strain of the ZnTiO₃ thin-film with respect to Oxygen Mixing Percentage (OMP) (Ar = 100 % to 0 %, O₂ = 0 % to 100 %).

Figure 2

The AFM surface micrographs of the ZnTiO₃ thin-films deposited at (a) 0 % (Ar = 100 % , O₂ = 0 %), (b) 12.5 % (Ar = 87.5 % , O₂ = 12.5 %), (c) 25 % (Ar = 75 % , O₂ = 25 %), (d) 50 % (Ar = 50 % , O₂ = 50 %), (e) 75 % (Ar = 25 % , O₂ = 75 %), (f) 100 % (Ar = 0 % , O₂ = 100 %) Oxygen mixing percentages.

Figure 3

Roughness of the ZnTiO₃ thin-film with respect to Oxygen Mixing Percentage (OMP) (Ar = 100 % to 0 %, O₂ = 0 % to 100 %).

Figure 4

(a). Transmittance spectra of ZTO thin-films deposited at (Ar = 100 % to 0 %, O₂ = 0 % to 100 %) Oxygen mixing percentages. (b). Tauc's plot of ZnTiO₃ thin-films deposited at thin-films deposited at (Ar = 100 % to 0 %, O₂ = 0 % to 100 %) Oxygen mixing percentages. (c). Refractive index (n_f), absorption coefficient (α), Extinction coefficient (K) and optical energy bandgap(E_g) of ZnTiO₃ thin-film with respect to the Oxygen mixing percentages.

Figure 5

EDS Spectra of ZnTiO₃ thin-film on N-type Si substrate a) the typical microstructure and elemental mapping for 25 Oxygen mixing percentage, b) the EDS Spectra of ZnTiO₃ thin-film for 25 OMP.

Figure 6

The Raman spectra of the ZnTiO₃ thin-films deposited at (a) 0 OMP, (b) 12.5 OMP, (c) 25 OMP, (d) 50 OMP, (e) 75 OMP, (f) 100 OMPs.

Figure 7

I-V Characteristics of ZnTiO₃ thin-film on N-Si substrate deposited at 25 OMP.

Figure 8

(a). $\ln(I)$ - V Characteristics of ZnTiO₃ thin-film on N-Si substate deposited at 25 OMP. (b). The bias-dependent resistance $R_i = dV/dI$ vs V Characteristics of ZnTiO₃ thin-film on N-Si substate deposited at 25 OMP. (c). V - $\ln(I)$ Characteristics of ZTO thin-film on N-Si substate deposited at 25 OMP.