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Diode production and characterization by evaporation of silver on ZnO thin films by cold substrate method

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ABSTRACT

In this study, ZnO (Zinc Oxide) thin films, which are widely used in photoelectronic technology, were grown on glass and tin oxide-coated glass substrates by chemical spraying at a substrate temperature of 325°C. The film growth rate was set at 60 Å per minute, while the spray rate was kept at 5 ml per minute. Thus, the thickness of the obtained ZnO films was approximately 1.8-2.1µm during 30 minutes of spraying. Then, the obtained ZnO films were annealed at 350 °C, 375 °C and 400 °C for 30 minutes at room temperature. The analyses were made on four different samples. X-ray diffraction patterns showed that all samples grew in a hexagonal crystal structure. It was determined from the Scanning Electron Microscope (SEM) images that the unannealed and annealed samples at 350 °C, 375 °C and 400 °C were in the form of nanorods and underwent a structural transformation with the effect of temperature. The energy band gaps calculated from the optical transmittance spectra of the samples were found to be 3.30, 3.25, 3.06 and 3.03 eV for unannealed ZnO and annealed ZnO films at 350 °C, 375 °C, 400 °C, respectively. Defects in the crystal structure of ZnO films were determined from the photoluminescence measurements. In the second stage of the study, the samples were divided into two groups and Ag was evaporated to their surfaces at substrate temperatures of 300 K and 200 K, and Ag/ZnO/SnO_2 binary structures were obtained. For the formation of Schottky diodes, the thermal diffusion process was applied to the binary structures produced at 300 K substrate temperatures for 30 minutes at 400 °C, while the photo-stimulated diffusion process was applied to the binary structures produced at 200 K substrate temperature for 1 hour at 375 nm wavelength under Ultraviolet (UV) rays. The barrier height and reverse saturation current values of these Schottky diodes, which were created through different technological applications, were determined and the results were compared.

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1. INTRODUCTION

Today, the contribution of studies on semiconductors to the rapid development of technology is undeniable. Semiconductor thin films are moving in line with technological developments. Thin films have been prepared using various methods since the 1950s. By definition, thin films are semiconductor materials, typically around 1 µm in thickness, where the atoms or molecules of the material to be coated using different manufacturing techniques are formed as a thin layer on a substrate [1].

Thanks to the developments in semiconductor technology, the devices obtained are reduced in size and increased in processing speed. Undoubtedly, nanotechnology has a great share in the development of electronic technologies. Semiconductor thin-film applications have significantly facilitated the production of electronic circuit elements and significantly reduced their size [2-6].

The methods which are widely used in the production of semiconductor thin films and which yield efficient results are the chemical sputtering method, thermal evaporation method, electron beam evaporation, sputtering method, and molecular beam epitaxy method. In this study, among these methods, the chemical spraying method and cold bridging technique, which is called innovative technology in thin film production technology, were used.

Singh and Park revealed in their study that dense ZnO nanorod films were deposited on an Indium tin oxide (ITO) substrate by hydrothermal method [4]. Structural and morphological properties of ZnO nanorod films were investigated using SEM and X-ray diffraction (XRD) devices. In the XRD scan, a high-intensity (002) peak was obtained, indicating the presence of normal *c*-axis oriented ZnO nanorod films on the ITO substrate. Ag/ZnO Schottky diode-based UV sensor was also produced. The characteristics of the samples (I-V) were investigated under dark and UV light in the voltage range from 0 V to +1 V. The nonlinear (I-V) nature of the UV photodetectors confirmed the Schottky contacts between Ag and ZnO junctions. The photocurrent and dark current contrast ratio of UV photodetectors produced under +1 V voltage was found to be approximately 1.67. Therefore, ZnO nanorod films were determined to have possibilities for applications in UV sensing devices.
In the study of Kucukömeroglu et al., the ZnO nanorod structure was produced by the chemical spraying method on SnO_2-coated glass substrates [7]. From the SEM and XRD results, it was determined that

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the ZnO nanorods had a hexagonal crystal structure and compressed rod morphology. Photoluminescence results showed a distinct emission peak corresponding to the UV region. Ag/ZnO nanorods/SnO_2/In-Ga characterized as a function of temperature by I-V measurements exhibited rectifying behaviour in the Schottky diode. It was observed that the ideality factor and barrier height values in the case of zero feed depended on the temperature. For ϕ_{B0} and A** values, which are consistent with the results of the studies, 0.88 eV and 2.75×10^{-5} A m^{-2} K^{-2} values were obtained.

In the study conducted by Kadhim and Ameer, ZnO nanoparticles were deposited on glass substrates by sol-gel method, and silver nanoparticles were deposited by laser ablation [8]. UV, SEM, and XRD analyses were used to characterize the prepared films. It was determined that the average ZnO particle size was 13.8 nm, and the Ag particle size was 12.4 nm [8].

In this study, ZnO thin films were grown on glass and SnO_2-coated glass substrates by chemical spraying at a substrate temperature of 325 °C. X-ray diffraction patterns showed that all samples grew perpendicular to the substrate along the c-axis in the hexagonal crystal structure. It was observed that the energy band gap values of the unannealed and annealed samples at different temperatures varied between 3.08 eV and 3.25 eV. Defects in the crystal structure of ZnO films were determined from the photoluminescence measurements. In the second stage of the study, the samples were divided into two groups and Ag was evaporated to their surfaces at substrate temperatures of 300 K and 200 K, and Ag/ZnO binary structures were obtained. For the formation of Schottky diodes, the thermodiffusion process was applied to the binary structures produced at 300 K substrate temperatures for 30 minutes at 400 °C, while the photo-stimulated diffusion process was applied to the binary structures produced at 200 K substrate temperature for 1 hour under UV rays. The barrier height and reverse saturation current values of these Schottky diodes created through different technological applications were determined.

2. EXPERIMENTAL PRODUCTION PHASE

In this study, pure and SnO_2-coated conductive glasses were used as substrates. Before starting the cleaning of the substrates, the samples were cut into 2 cm x 3 cm dimensions and then cleaned in an ultrasonic bath with distilled water for 5 minutes, ethanol for 5 minutes, and distilled water for 5 minutes again. The substrates were dried at 70 °C for 1 hour and made ready.

To prepare 0.15 M ZnO solution, 5.11 g of zinc chloride salt ($ZnCl_2$, Aldrich, 98% purity) was added to 250 ml of purified water according to the following chemical reaction.

$$ZnCl_2 + H_2O \Rightarrow ZnO + 2HCl$$

To prepare the mother solution, 5.11 g of $ZnCl_2$ salt was weighed through a precision electronic balance. The resulting solution was mixed using a magnetic stirrer for 20 minutes at 70 °C. The prepared solution was sprayed onto glass surfaces heated at 325 °C. The litter temperature was monitored using a chromium-alumina thermocouple. The distance between the spray nozzle and the substrate was set to 50 cm, the spray angle was set at 60˚, and the spray speed was set at 5 ml/min. In this application, the thin film growth rate was maintained at 50 Å per minute.

To determine the behaviour of the crystal structures of the obtained ZnO thin films depending on the temperature, the samples obtained on the surface of the glass and SnO_2 substrates were annealed for 30 minutes at three different temperature values (350 °C, 375 °C and 400 °C) in a room environment. The crystal structures and phases of the ZnO films prepared with the help of an X-ray diffraction device were determined. Scanning electron microscopy was used to determine the surface properties of the samples. The optical and photoluminescent properties of the films were characterized by photoluminescence spectroscopy and spectrophotometer.

Ag was evaporated under a high vacuum (10^{-5} Torr) with the help of a Vaksis PVD handy/1DLE-LN model device. Evaporation processes were performed at two different substrate temperatures (300 K and 200 K). While Ag was evaporated onto the surface of ZnO thin films, a mask with a diameter of 1 mm was used. Thus, it was ensured that the obtained diodes were of equal area.

In Figure 1, a schematic view of the steps performed in the cold substrate coating method is given. The Ag metal evaporated in the 1st step is sublimated by heating and saturated vapour of the metal is formed in the reactor section of the apparatus. During this process, the window is kept closed and the litter material is cooled with liquid nitrogen. In the second stage of the application, the window is opened and the substrate cooled with saturated steam is brought into contact. During the contact of two different (heat and cold) environments, soliton waves are formed on the substrate surface. In the third stage, metallic nano-sized clusters transported from the saturated vapour environment to the cooled substrate surface are arranged homogeneously by soliton waves.

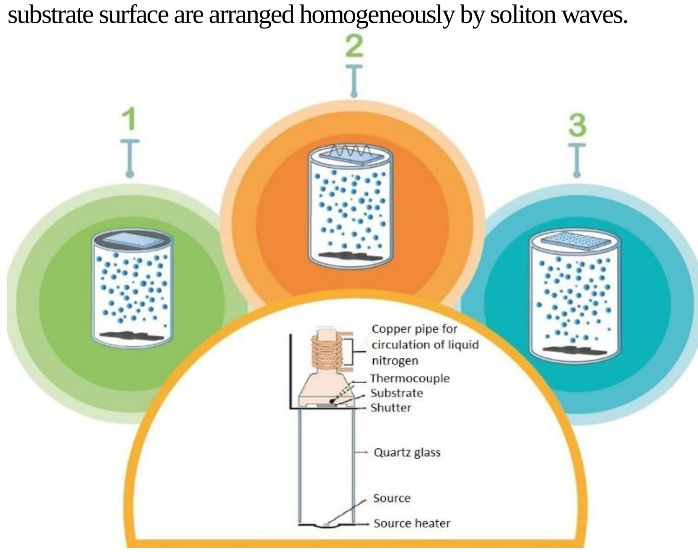


Fig 1. Schematic representation of the coating steps performed by the cold substrate method.

The biggest advantage of the cold substrate (200 K) method and its difference from other methods is the formation of a soliton wave during the collision of the clusters, which are transported to the substrate surface from the saturated vapour and positioned here, with the hexagonal atoms. Non-linear soliton waves allow particles to move on the substrate surface without losing energy due to their mass-carrying feature. Clusters that realize the soliton growth mechanism with the formation of a soliton wave are called critical-sized clusters. Critical-sized clusters vary between 10 -15 nanometers depending on the temperature of the substrate surface.

The following inequality must be satisfied for critical-sized clusters to form a soliton wave on the substrate surface in Equation 1.

$$\frac{a(T_r)-b(T_f)}{b(T_f)} > \left(\frac{2}{\pi}\right)^{3/2} \sqrt{\frac{f}{\lambda} a(T_r)} \qquad (1)$$

a(Tr) and $b(T_f)$ are the lattice parameters of the substrate and the incoming particle, respectively. *f* indicates the interaction force between the substrate and the cluster (all clusters from saturated vapour) and *λ* the interaction force between the substrate atoms [1].

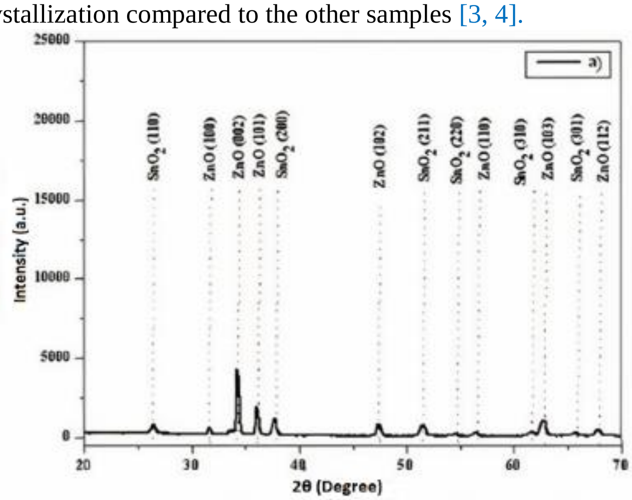
While the thermal diffusion process was applied to the binary structures produced at 300 K substrate temperature for 30 minutes at 400 °C, the photo-stimulated diffusion process was applied to the binary structures produced at 200 K substrate temperature under UV rays at a wavelength of 375 nm for 1 hour. For the formation of the gate in Ag/ZnO binary structures obtained at a substrate temperature of 200 K, a UV light source, which is used for the realization of the photo-stimulated diffusion process, was used.

3. RESULTS AND DISCUSSION

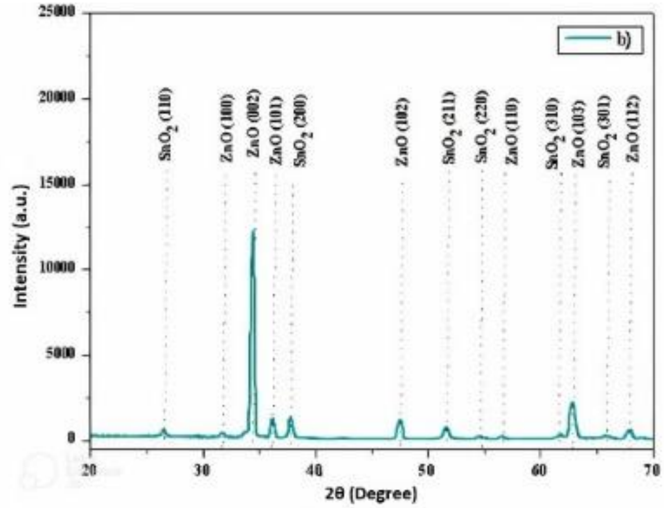
X-ray diffraction results were recorded at room temperature (Cu-Kα) at 40 kV and 30 mA with the help of wavelength λ= (1.54 Å). Scanning was done in continuous mode, and the step width was kept at 0.02°. The X-ray diffraction results of ZnO thin films are shown in Figure 2 (a-d).

The XRD patterns showed that the produced films grew in the (002) plane in the c-axis direction. In addition, reflections arising from any other impurities were not encountered in these diffraction patterns because of annealing.

In Figure 2, the Bragg angles are 31.68°, 34.442°, 36.16°, 47.43°, 56.44°, 62.745°, 67.94° (100), (002), (101), (102), (110), (103). Reflections are seen from (201) planes. According to the XRD card numbered (JCPDS 36-1451), it was determined that ZnO thin films had a wurtzite (hexagonal) structure. However, as the annealing temperature increased, it was observed that the reflection intensities of all diffraction peaks increased. As can be seen from the XRD patterns, the 400 ºC annealed ZnO sample shows the highest reflection intensity [2-5]. As a result of this, it is understood that this sample has a higher degree of crystallization compared to the other samples [3, 4].



(a)



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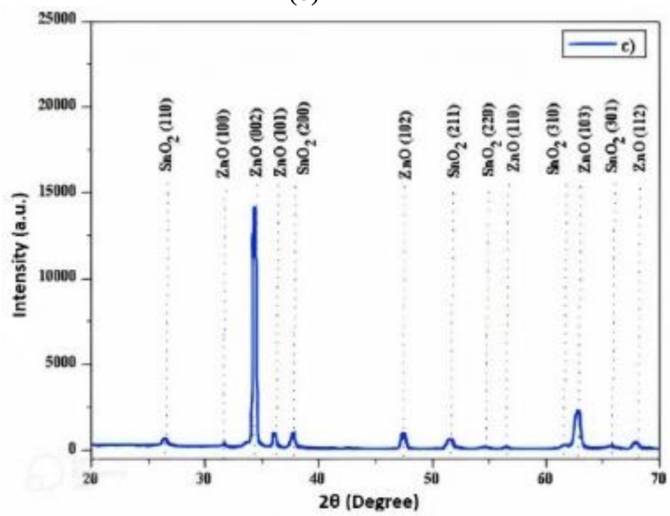

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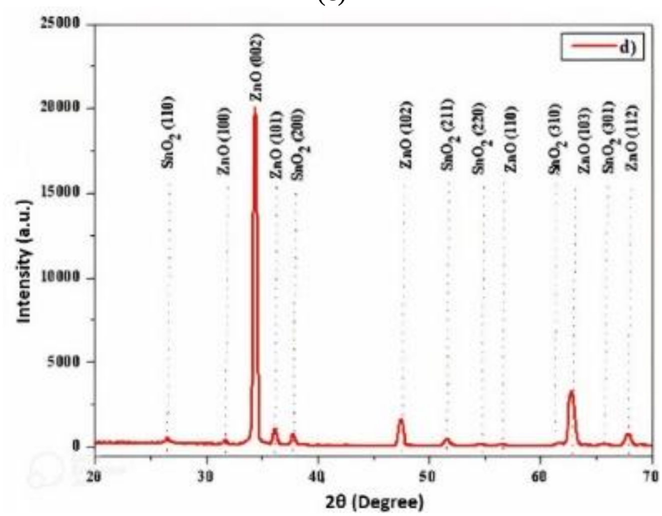
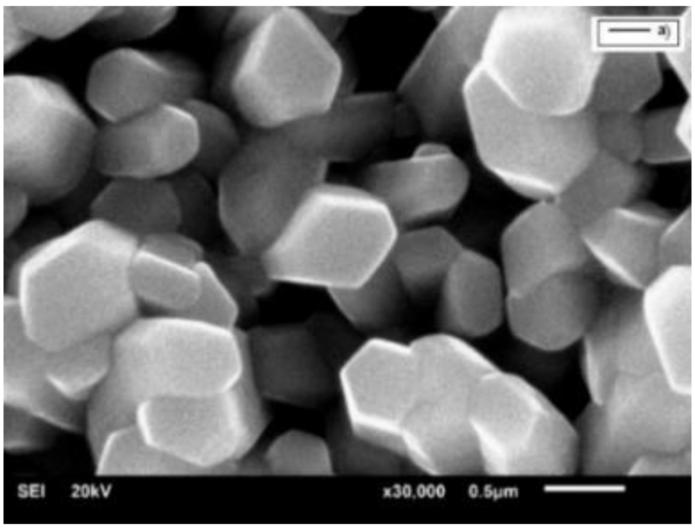


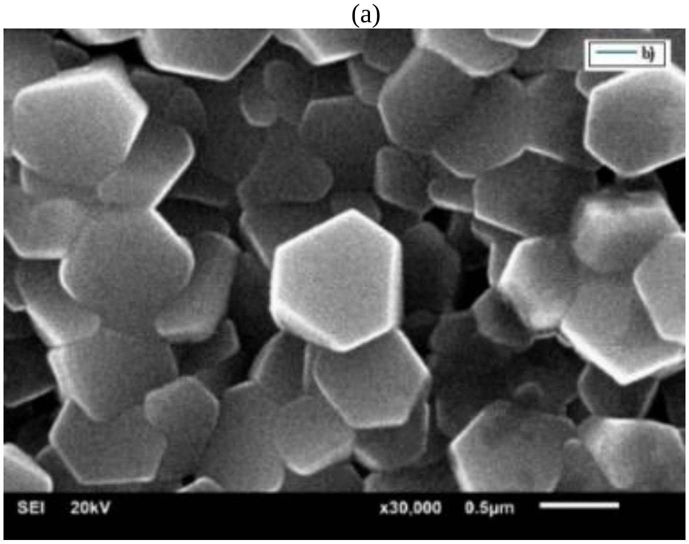
(d)

Fig. 2. X-ray diffraction of ZnO thin film grown on SnO_2 coated glass surface and annealed at different temperatures, (a) Unannealed sample, (b) 350 ˚C, (c) 375 ˚C and (d) 400 ˚C annealed samples.

Scanning electron microscope images of ZnO films prepared by the sputtering method on the glass surface are given in Figure 3. It is understood that the films obtained from SEM images have a porous surface morphology. The images show that the pure ZnO thin film has a microrod structure [4,5]. In addition, it is seen from the SEM images that there is an increase in the width of the ZnO bars with the annealing effect, and as a result, the gaps between the micro rods become smaller [6-9]. In addition, as the annealing temperature increases, homogeneity begins to occur on the produced ZnO surfaces. This result is in line with the increase in crystal bar width expected from annealing and the prediction of a more regular film [10]. This result coincides with the XRD results and is consistent with the results of the study by Aljwafi et al. [11]. In the Aljwafi study, ZnO nanoparticles were produced by sol-gel method and annealed at different temperatures (200 °C, 400 °C, 600 °C and 800 °C). It was reported that the grain size increased with the increase in annealing temperature [11-13].



(a)



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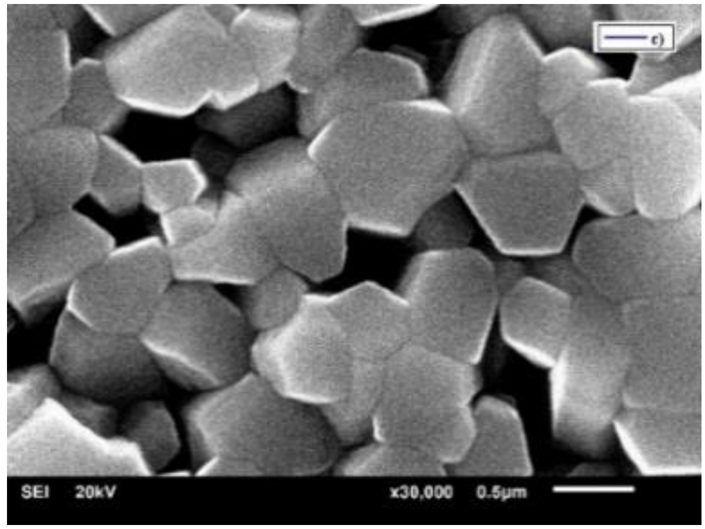

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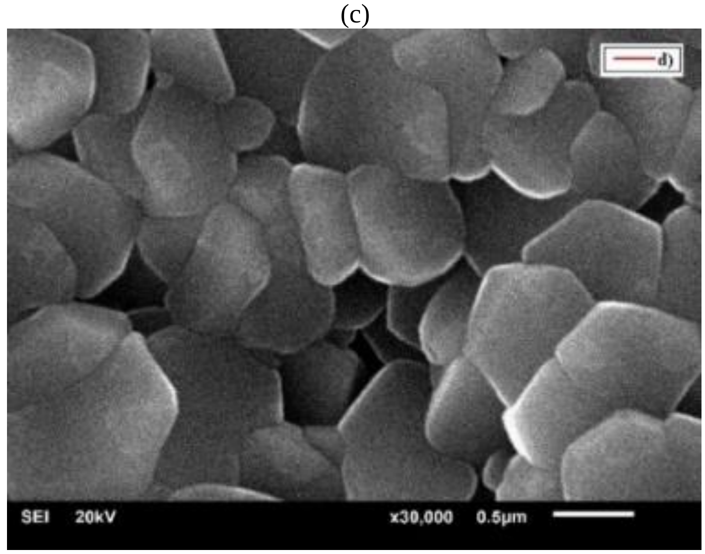


(d)

Fig. 3. SEM image of the produced ZnO thin film, (a) unannealed sample, (b) samples annealed at 350 ˚C, (c) 375 ˚C, (d) 400 ˚C.

Optical transmittance measurements were made at (200-800) nm wavelengths, at room temperature, with the help of Spectra brand M5 model photoluminescence spectroscopy device. In Figure 4, the optical transmittance spectra of ZnO thin films are given. As can be seen from the figure, the optical transmittance increases with increasing annealing temperature. This phenomenon can be explained by the fact that the samples have better crystallization and lower defect density with increasing annealing temperature [14].

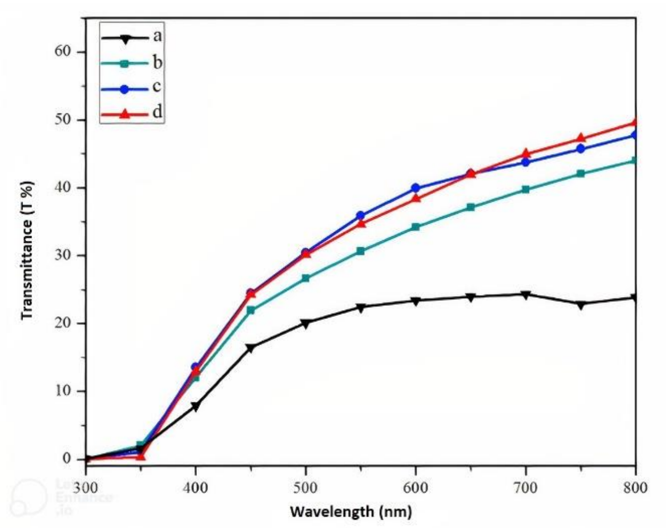


Fig. 4. Transmission spectra of ZnO thin films, (a) unannealed sample, (b) samples annealed at 350 ˚C, (c) 375 ˚C, (d) 400 ˚C.

Equation 2 was used to determine the forbidden band gap.

$$\alpha hv = A(hv - E_g)^{1/2} \quad (2)$$

Here Eg is the forbidden band gap of the sample, and A is a parameter that depends on the refractive index of the material. Figure 5 gives a graph of $(\alpha hv)^2$ as a function of photon energy (hv). The point where the linear part of this graph intersects with the x-axis gives the value of the direct band energy gap.

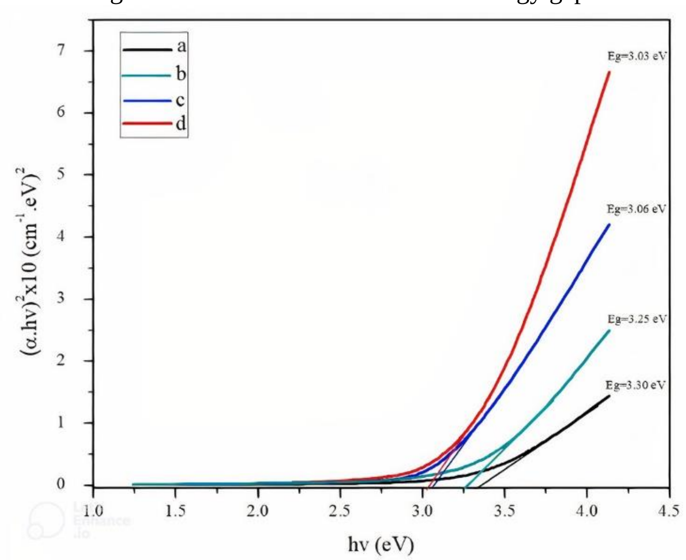
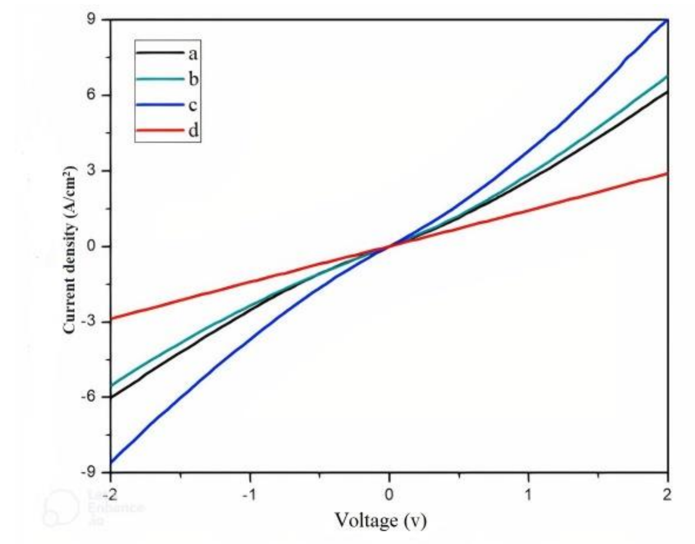


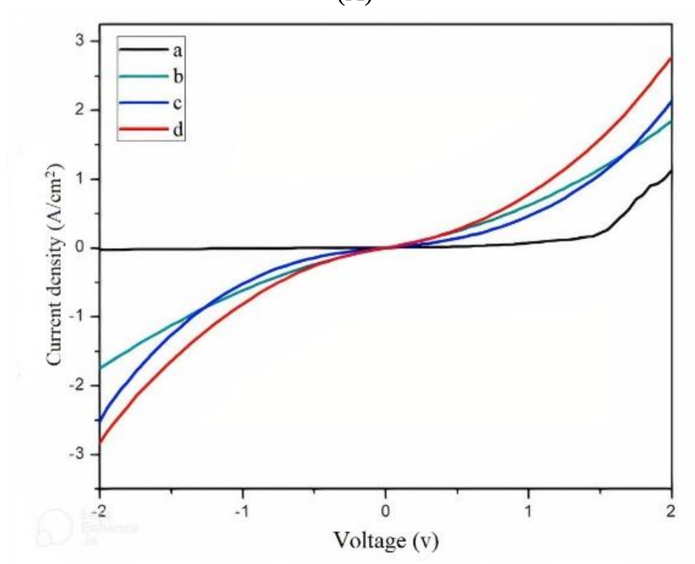
Fig. 5. Representation of the $(\alpha hv)^2$ curve of the produced ZnO thin films according to the photon energy, transmittance spectra of the ZnO thin films, (a) unannealed sample, (b) 350 ˚C, (c) 375 ˚C and (d) samples annealed at 400 ˚C.

In Figure 5, the energy band gaps of unannealed ZnO and annealed ZnO films at 350 ºC, 375 ºC, and 400 ºC were found to be 3.30, 3.25, 3.06 and 3.03 eV, respectively. The energy band gap for semiconductors was in the range of 0.5 to 5 eV, and for transparent oxide semiconductors, it was around 3-5 eV. Thus, it was seen that the forbidden band values found were in harmony with the standard values. It was observed that the energy band gap values decreased with increasing annealing temperature. These results can be explained by the increase in the width of the ZnO bars seen in the SEM graphs with the increase in the annealing temperature [14, 15].

In Figure 6, the measured J-V properties of Schottky diodes formed by two different methods are given in the forward and feedback states.



(A)



(B)

Fig. 6. Characterization of Schottky diodes (J-V) produced at (A) 300 K, (B) 200 K substrate temperature and subjected to the photoinduced diffusion process. (a) unannealed $(SnO_2/ZnO)/Ag$, (b) 350 ˚C, (c) 375 ˚C and (d) annealed $(SnO_2/ZnO)/Ag$ at 400 ˚C.

In Figure 6 (A), the sample of $SnO_2/ZnO/Ag$ produced at a substrate temperature of 200 K and subjected to a photo-stimulated diffusion process without annealing the base layer (ZnO) shows a rectifying property suitable for the diode characteristic. In Figure 6(B), it is seen that the samples produced at a substrate temperature of 300 K and subjected to the thermo-stimulated diffusion process do not have rectifier properties. This behaviour can be explained by the presence of surface defects of the microrod films of zinc oxide or by barrier inhomogeneity at the metal/semiconductor interface. As can be seen from Figure 6, the (Unannealed SnO_2/ZnO)/Ag diode produced at a substrate temperature of 200 K allows current to flow at 1.25 volts and shows a late break under reverse supply, but all other generated diodes become active at 0.1 volts and under reverse supply.

The direct current of the diode can be calculated in Equation (3).

$$I = I_o exp\left(\frac{qv}{2nkT}\right)\left[1 - exp\left(-\frac{qv}{2nkT}\right)\right] \quad (3)$$

Here V is the applied voltage, q is the electron charge, and I_0 is the reverse saturation current. The saturation current (I_0) values are found by extrapolating the linear region of the I-V curves to zero applied voltage in the semi-logarithmic feed-forward condition and are expressed by the following Equation (4).

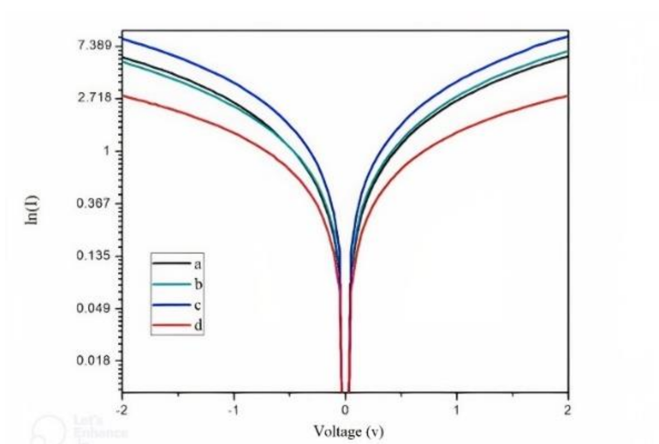
$$I_o = AA^{*}T^2 e^{\phi_b/kT} \quad (4)$$

Here A^* is the effective Richardson constant (32 A/cm^{2}K^2 for n-type ZnO), A is the effective diode area, T is the temperature in kelvin, k is the Boltzmann constant, and ϕ_b is the effective barrier height.

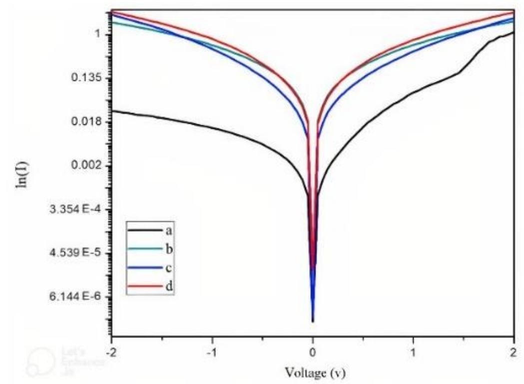
It is important to examine the ln(I)-V characteristic to calculate the electrical parameters of the diode. In Figure 7(B), using the experimental results, the ln(I)-V characteristic for the diodes produced at a substrate temperature of 300 K is given. In Figure 7(A), using the experimental results, the ln(I)-V characteristic for the diodes produced at the substrate temperature of 200 K is given.

By determining the saturation current value, the barrier height was calculated with the following Equation 5.

$$\phi_{bo} = \frac{kT}{q}\ln\left(\frac{AA^{*}T^2}{I_o}\right) \quad (5)$$



(A)



(B)

Fig. 7. (A) 300 K substrate temperature, (B) 200 K substrate temperature of the Schottky diode (lnI-V) graph.

The electrical parameters of SnO_2/ZnO/Ag Schottky diodes produced at different substrate temperatures are given in Table 1.

According to this study, the interface states depend on different factors such as metal properties, deposition process, surface treatment, and density of surface defects. It is seen from Table 1 that the reverse saturation currents of the diodes produced at 200 K substrate temperature are lower than the reverse saturation current values of the diodes produced at 300 K substrate temperatures. As can be seen from Table 1, the barrier height of the diodes produced at 200 K substrate temperature is higher than the barrier height values of the diodes produced at 300 K substrate temperature. This situation may be due to the existing surface conditions, the height of the barrier, and the effect of the oxide layer, together with the difference in the material and method used at the beginning [17, 19]. Especially because ZnO structures exhibit high optical properties, it is used as an optical filter in thin-film solar cells [20]. As can be seen from the table, the barrier heights of Schottky diodes produced at 200 K decreases as the annealing temperature increases. In addition, there is a slight increase in 300 K. As it is known, the barrier height changes depending on the series resistance values [21]. As the annealing temperature increases, oxygen penetrates the deeper layers, causing an increase in the series resistance in the ZnO structure. Considering that the diode produced at 200 K is in the form of a tight package, it is understood that the diffusion coefficient of oxygen atoms is lower compared to 300 K. Another result that supports these results is the increase in current values.

Table 1. Electrical parameters of Schottky diodes fabricated at 200 K and 300 K substrate temperatures.

Substrate Temperature (K)	*ZnO Annealing temperature (°C)*	*I_0(A)*	*Φ_b (eV)*
200	Unannealed	4,7 x 10^{-6}	0,57
	350	4,8 x 10^{-5}	0,51
	375	4,2 x 10^{-5}	0,52
	400	6,6 x 10^{-5}	0,50
300	Unannealed	4,8 x 10^{-4}	0,45
	350	4,0 x 10^{-4}	0,46
	375	7,3 x 10^{-4}	0,44
	400	4,2 x 10^{-4}	0,46

4. CONCLUSION

ZnO films were produced by a chemical spraying method, the obtained ZnO films were annealed at 350 ºC, 375 ºC and 400 ºC temperatures for 30 minutes in a room environment. The analyses were made on four different samples. X-ray diffraction patterns showed that all samples grew in a hexagonal crystal structure. In addition, many different oxide phases belonging to the ZnO structure are seen in the XRD spectra due to the growth of the films in the room environment. Increases in nano-bar widths of unannealed and annealed ZnO samples at different temperatures were detected from SEM measurements. From the optical transmittance measurements recorded in the wavelength range of 200–800 nm, it was observed that the ZnO thin film annealed at 400 ºC had higher optical transparency. This is due to the higher crystallization intensity as seen in the XRD spectra. It was observed that the energy band gaps (Eg) calculated from the optical transmittance spectra of the samples varied between 3.03 eV and 3.3 eV. The results obtained are due to decreases in the density of quantum energy levels due to oxygen vacancies in the forbidden band gap during annealing. In the second stage of the study, the samples were divided into two groups and Ag was evaporated to their surfaces at substrate temperatures of 300 K and 200 K. For the formation of Schottky diodes in the obtained Ag/ZnO binary structures, a thermal diffusion process was applied to the binary structures produced at 300 K substrate temperature, called the first group, at 400 ºC for 30 minutes. Photo-stimulated diffusion process was applied to the binary structures produced at a substrate temperature of 200 K, called the 2nd group, under UV rays at a wavelength of 375 nm for 1 hour and Schottky diodes were formed. With electrochemical measurements, it was determined that the barrier height of Schottky diodes produced in Ag/ZnO binary structures at 200 K substrate temperature was higher than 300 K. It has been understood that the differences in barrier height are due to the narrower displacement region formed at 200 K.

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