The characterization of amorphous AZO-n/Si-p hetrojunction diode for solar cell application

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Abstract
The aim of the present study is to verify the effect of annealing temperature variation on zinc oxide doped with aluminum thin films deposited on p-type silicon substrates. Here, all thin films were annealed in nitrogen environment and its structural, electrical, and optical characterizations were investigated. The results of X ray diffraction patterns showed the amorphous structure of zinc oxide doped with aluminum thin films. Field Emission Scanning Electron Microscopes images illustrated the increase of grain size by increasing annealing temperature up to 500 °C. The reflectance analysis showed that for this annealing temperature, the energy band gap of zinc oxide doped with aluminum thin film was moved to higher energy level. The electrical properties were investigated by Current–Voltage measurement carried out in the light at room temperatures. The short circuit current, ideality factor, saturation current, and open circuit voltage of the zinc oxide doped with aluminum on silicon heterojunction strongly depended on annealing conditions due to charge carrier trapping and density of defect on interface. By considering reverse current and forward current, the ratio of forward current to reverse current had the maximum value at 1 V which was belonged to n type zinc oxide doped with aluminum on p type silicon heterojunction at 500 °C annealing temperature.

Keywords AZO-n/Si-p heterojunction · Amorphous · Annealing conditions · Sputtering procedure

1 Introduction

Transparent conducting oxide (TCO) films have attracted a lot of attention in recent decades. Zinc oxide is a good candidate for TCO films due to excellent electrical, structural, and optical properties like doping suitability and non-toxicity, as well as high thermal, mechanical, and chemical stability (Sahay and Nath 2008; Dejam et al. 2015). Therefore,
ZnO can be considered as suitable choice in optoelectronic and electronic devices like, gas sensors, transparent electrodes, light emitting diodes, varistors, nanolasers heterojunctions, and etc. (Wang et al. 2007; Kluth et al. 2003) Moreover, ZnO thin film deposited on p/n silicon substrate can be applied in optoelectronic devices and solar cells as a useful heterojunctions whose main advantages are the low cost of silicon and the high energy of band gap of ZnO thin films (Breivik et al. 2007).

Because of high sensitivity of pure ZnO thin films to the oxidation and also its high transmittance in the visible area, ZnO thin films were doped with aluminum initially. Then, all samples were annealed in order to decrease resistivity and increase band gap energy, carrier concentration, and stability (Ilkhani and Dejam 2021). On the other hand, Al:ZnO (AZO) is a promising material for light emitting diodes, solar cells, and gas sensors along with general optoelectronic applications (Al-Hardan et al. 2016; Dejam et al. 2017).

The advantages of silicon-based solar cells are like non-toxicity and low cost make them to be applied in photovoltaic systems (Liu et al. 2017). AZO thin films as a front electrode must be conductive enough to allow lateral transfer of electron from sufficient light (80%) to the incoming light (Dejam et al. 2016).

Several methods have been applied to fabricate AZO thin films including pulsed laser deposition (PLD), evaporation, chemical vapor deposition, magnetron sputtering, and spray pyrolysis (Lu et al. 2009; Dejam et al. 2019). Among these methods, thin films with high-quality are prepared by magnetron sputtering technique because of its special conditions like low temperature deposition, large-area fabrication adaptability, repeatability, and uniformity as well as working power and sputtering pressure (Talu et al. 2016).

Specific interest of ZnO/Si and AZO/p-Si heterojunctions is observed in the optoelectronic devices strong adhesion. So far, a lot of researches have been focused on AZO/p-Si heterojunction. Bo et al. (2009) fabricated Al-doped n-ZnO/p-Si heterojunctions by the deposition of AZO film on p-Si (1 0 0) wafer and investigated their optical, microstructural, and electrical properties. They found good quality of samples and their rectifying behavior under dark condition. Urper et al. (2019) also fabricated Al:ZnO thin film by sol–gel dip coating method and studied the relationship between Al content and optical band gap of the films. They also analyzed crystalline structure and electrical resistivity of samples under different annealing conditions. Baydogan et al. (2012) found hexagonal wurtzite crystal structure of ZnO:Al thin films prepared by sol–gel dip coating method. They also demonstrated that the optical band gap was broadened by increasing Al concentration. The novelty of our work is that we have focused on the optical, structural and electrical properties of amorphous AZO thin films prepared by sputtering. So far, according to our studies, amorphous AZO thin films has not been considered as a diode and making amorphous thin films is easier and cheaper. Also we have investigated the improvement of amorphous AZO/p-Si heterojunction and find the best structural conditions for AZO/p-Si as a diode and solar cell.

In the present work, AZO thin films were deposited on p type silicon substrate by RF magnetron sputtering method. AZO thin films were annealed with nitrogen gas at three different temperatures. Then, their electrical, optical, and structural properties as well as their morphology were investigated. For this purpose, every junction of AZO amorphous thin film and Si was taken into consideration and Si/AZO heterojunction diode properties were determined. As can be seen, annealing in nitrogen atmosphere and Al dopant ends to the extreme enhancement of blue, violet and green emissions.
2 Experimental details

Zinc oxide doped with aluminum thin films (AZO) were deposited by radio-frequency (RF) magnetron sputtering on p-type silicon (Si) substrate. The diameter of circle-shaped target was 2.5 cm with the thickness of 3 mm. The weight ratio of Al and Zn were ~10% and 90%, respectively. The sputtering gas was constant amount of (O$_2$ + Ar) where the ratio of O$_2$ was 30% and the power of sputtering was 175 W. The basic pressure was set as $6.5 \times 10^{-5}$ mbarr. The substrates were initially cleaned and then placed in acetone and ethanol ultrasonic bath for 15 min. They were finally dried in a clean room. In order to remove any oxide residues, samples were pre-sputtered 30 min. Afterward, samples were annealed for 60 min at 400 °C, 500 °C and 600 °C temperatures at the presence of nitrogen gas and then, they were cooled down to room temperature gradually. The details of sputtering process are presented in Table 1. The thickness of prepared AZO thin film was measured 200 ± 5 nm.

To characterize the crystalline nature of samples, X-ray diffraction (XRD) patterns were extracted from STOE-XRD diffractometer using Cu-Kα line ($\lambda = 0.15406$ nm). Also, the topography of AZO thin films was investigated by the non-contact mode atomic force microscopy (AFM) (Vecco-Autoprobe-research) and field emission scanning microscopy (FESEM) (MIRA3 TESCAN). Energy dispersive X-ray (EDX) analysis was performed by MIRA3 TESCAN to analyze the information about the elements of samples. The optical transmittance spectra of the deposited films were recorded by the UV–VIS–NIR spectrophotometer (CARY-500 UV–VIS-NIR) in the range of 200–1600 nm. Photoluminescence (PL) was also used to reveal the luminescence characteristics of samples by Cary Eclipse spectrometer equipped with a xenon amp with 320 nm (AZO) excites wavelength. The current–voltage (I-V) measurement was finally carried out by solar simulator (SIM-1030) and Palm Sense.

3 Results and discussion

3.1 Structure properties

Figure 1 illustrates XRD patterns of AZO thin films on silicon substrates. As can be seen, there is just one peak at 68 degree related to silicon substrate and there is no peak related ZnO (hexagonal wurtzite) crystal structure. Therefore, amorphous nature of AZO thin films is demonstrated by XRD patterns. The crystalline peak of Si is of very high intensity. Therefore, the AZO thin films on the glass substrate were also grown and annealed under the same conditions to examine their XRD spectrum, which may indicate ZnO crystalline peaks. But no specific peak was observed for the crystal structure of ZnO on the glass substrates. The method of preparing the AZO thin films can be the reason for the amorphous structure of the thin films. Existence of Zn/Al target and oxygen gas enters the environment during sputtering. For thin thicknesses, it is not possible to form the crystal structure of AZO thin films. In the same manufacturing and annealing conditions with thicker thicknesses, we prepared the crystal structure of AZO thin films (Dejam et al. 2016; Talu et al. 2016). Similar results have been gained by other researchers who prepared AZO thin films without annealing process (Dejam et al. 2017, 2016; Liu et al. 2017). Incidentally, the interesting point of the research is that although there is no change in thin film structure up to 600 °C annealing temperature, the electrical and optical properties of samples have
been changed. Most research focus on the amorphous nature of AZO thin films. However, their crystalline structure has been studied extensively (Bo et al. 2009; Urper et al. 2019; Baydogan et al. 2012).

As can be seen in Fig. 2a–d, EDX data approved the existence of Zn and Al in as-deposited and annealed thin films and determined their stoichiometry. Moreover, any significant variation were observed in weight and atomic percentage of AZO thin films after annealing process.

FESEM images in Fig. 3a–d represent the topography of as deposited and annealed AZO thin films. As can be seen, there are irregular and non-uniform nanograins in as deposited sample whose average size is ~ 55 nm. Increasing annealing temperature up to 500 °C increases the average diameter because of agglomeration and the best grain adhesion is occurred at 500 °C and the grain boundaries are much smaller, which is very important for electrical properties because they control the movement of electrical charges. By increasing annealing temperature to 600 °C, the distribution is disrupted due to grain movements. In fact, at this temperature, the particles have not become smaller, but have dispersed, and the grain boundaries have increased. This is also confirmed in the AFM particle distribution diagram (Table 2).

Surface morphology of AZO films was examined by their 3-D AFM images (a–d) as illustrated in Fig. 4. As can be seen, surface roughness is increased by increasing annealing temperature (Table 2) which may be due to the grains stickiness, the creation of a rough surface, and the variation of RMS roughness under nitrogen gas flow range from 0.42 to 1.61 nm. In addition, the surface of as-deposited AZO thin film is smoother and more interconnected which is formed by annealing particles at the grain boundaries, resulting in a variety of sizes. Figure 5 shows the morphological changes of thin film surface. Particle distributions differ from the Gaussian diagram by increasing annealing temperature. Especially at 600 °C, the dispersion of nanoparticles is much higher which is exactly the behavior observed in FESEM images. Increasing annealing temperature increases the energy and surface mobility which results in particle size enhancement. Enhancement in thermal and kinetic energy has also increased the dispersion of particles on the surface which is a key factor in changing the shape of the Gaussian distribution. On the other hand, increasing the size of particles and their dispersion on the surface is because of the adhesion of nanoparticles to each other.

### 3.2 Optical properties

Figure 6 indicates reflectance spectra of as deposited and annealed AZO thin films. In the reflectance spectra, a sharp band edge of about 350–400 nm in the as deposited and 400, 600 °C annealed thin films is due to strong adsorption. An obvious blue shift is only observed in AZO thin film annealed at 500 °C which is because of the suffering of its...
absorption edge. Reflection of AZO thin film at 550–800 nm decreases with annealing. Reduction of the reflection spectrum after annealing may be due to the nano-scaled morphology of the sample which has improved the light traps.

The UV–Vis spectra were inquired by Kubelka-Munk theory to convert thin film reflectance to Kubelka-Munk function (F(R)), by Eq. (1) (Williamson and Hall 1953; Baranski et al. 1983; Choudhary and Chauhan 2016):

\[ F(R) = \frac{(1 - R)^2}{2R} \]

where \( R \) is the reflectance of sample and depends on wavelength. Equation (2) shows the relationship between F(R) and the absorption coefficient (\( \alpha \)) as (Williamson and Hall 1953):

\[ \alpha = \frac{F(R)}{t} \]

where ‘t’ is the thickness of AZO thin films. To evaluate the dependency of band gap to direct allowed transition, the diagram of \((\alpha h\nu)^2\) vs. \( h\nu \) is illustrated in Fig. 7 and band gap was calculated via the linear \( h\nu \)–intercept. The value of band gap increases gently from 3.2 to 3.9 eV by increase of annealing temperature up to 500 °C. But when the annealing temperature increases to 600 °C, the value of band gap decreases. Hence, a critical state has occurred at 500 °C which is also confirmed in previous research (Dejam et al. 2016). In fact, annealing process decreases the defects while increasing temperature reduces the density of the localized states and decreases their band tail energy. Here, increasing annealing temperature to 500 °C decrease the density of the localized states and defects and increases the band gap.

However, AFM and FESEM images of sample annealed at 600 °C indicate surface irregularity and uneven distribution of nanoparticles and hence, enhancement of defects replacement and localized states as well as decrease of band gap are expected. It should be noted that results of PL analysis confirms these changes in defects, localized states, and band gap.
Fig. 2  EDX spectra of a as deposited, and annealed AZO/p-Si heterojunction at \textbf{b} 400 °C, \textbf{c} 500 °C, and \textbf{d} 600 °C
PL spectra in Fig. 8 are applied at room temperature to estimate any annihilation or defect created by annealing. PL emission proved the center of defects that act as centers of recombining charge carrier. For as-deposited and annealed AZO thin films excited at
320 nm, the PL spectra show four emission bands at UV, Violet, Blue and Green emissions. Fitting PL spectra with Gaussian diagram gives valuable information about FWHM and peak positions as summarized in Table 3. The peak at around 374 nm corresponds to UV emission or to the transition of electron from the substitution level below the conduction band to the valence band (Gao et al. 2004). The near band-edge emission with the peak at around 393–398 nm results in UV emission because of its dependency to the recombination of free-exciton (Zeng et al. 2010). The peak around 400 nm in all samples is a violet emission which appears because of the electron transition from conduction tail states to the valence states (Cho et al. 2010). Moreover, the blue emission observed around
(467–489 nm) is revealed by the transition from shallow donor levels of oxygen vacancy to valence band which is in agreement with Xue’s report (Xue et al. 2002). The peak at around (528–534 nm) belongs to green emission which is appeared by the transition from deep donor levels of oxygen vacancies to valence band (Liang et al. 2011). Annealing in nitrogen gas flow also affects the presence of green emission (Greene et al. 2003). With increasing annealing temperature up to 500 °C, the PL centers remarkably increase which are related to the increment in PL intensity. Therefore, increasing density of defects at 500 °C results in enhancement of recombination and hence, hence, the increase of $V_{oc}$ that can be seen in Table 2 where the maximum $V_{oc}$ is revealed in 500 °C.

### 3.3 The heterojunction properties of AZO/p-Si

In order to find remarkable information about Schottky diode behavior, current–voltage I-V diagram were measured under illumination at room temperature and presented in Fig. 9a for all samples. An almost photoelectric and rectifying behavior is illustrated in n-AZO/p-Si heterojunctions. Figure 9b also illustrates the semi-logarithmic forward and reverse bias of the experimental I–V for the n-AZO/p-Si Schottky diodes.

By considering $I_R$ and $I_F$ for reverse and forward current, the rectification $I_F/I_R$ ratio at 1 V is in the range of 0.38 to 1.42. Based on p–n junction theory, the I–V relation of standard diodes is as follow (Baydogan et al. 2012; Ghaderi et al. 2011):

$$I = I_s \exp \left( \frac{qV}{nk_BT} - 1 \right)$$  \hspace{1cm} (3)

where $I_s$ and $V$ is saturation current and voltage bias, respectively. Saturation current ($I_s$) is measured from the straight line intercept of Ln I at V = 0 as (Rezaee et al. 2014):

$$I_s = A^*ST^2 \exp \left( \frac{q\varphi_k}{k_BT} \right)$$  \hspace{1cm} (4)

Here, $q$ and $T$ are the absolute value of electron charge ($1.6 \times 10^{-19}$ C) and temperature, respectively. Also, $V$, $I$, and $S$ are applied voltage, net current, and diode area, respectively.
The Boltzmann’s constant ($k_B$) is $1.38 \times 10^{-23}$ J/K and the barrier height (eV) is labeled as $\phi_b$. Also, the Richardson constant ($A^*$) for ZnO is 32 A cm$^{-2}$ K$^{-2}$ (Singh et al. 2011). The ideality factor ($n$) of heterojunctions is calculated in Eq. 5 from the slope of the straight line in the forward bias $\text{Ln} I–\text{V}$ diagram (Baydogan et al. 2012)

$$n = \frac{q}{k_B T} \left( \frac{dV}{d\text{Ln}I} \right) \quad (5)$$

The values of $I_s$ and $n$ are represented in Table 2. The saturation current for $n$-AZO/p-Si heterojunction was also reported by for Urper et al. (2019) and Baydogan et al. (2012) as $1.5 \times 10^{-6}$ A and $0.3 \times 10^{-6}$ A, respectively and they are comparable to the value of $I_s$.
in the present study. As it is known, the ideality factor for an ideal diode is unity and for other devices is bigger than one. Here, the value of $n$ is similar to the previous research as 38 (Baydogan et al. 2012) and 20.1 (Bo et al. 2009) which demonstrates that present diode is not ideal and reason is the existence of surface states and oxide layer (Majumdar et al. 2009).

In order to find the response of photoelectrical parameters, open circuit voltage ($V_{OC}$) and short circuit current ($I_{SC}$) were calculated for AZO/p-Si Schottky diodes under 1000 W/m² of light source for as deposited and annealed AZO/p-Si heterojunctions. The values of these two parameters have been measured by results of I-V diagram in Table 2. The amounts of $I_{SC}$ and $V_{OC}$ are in the regions from 0.23 to 1.84 µA and 0.05–0.13 V, respectively. The adsorption results also show maximum and minimum values of $I_{SC}$ at AZO/p-Si annealed at 500 °C and as-deposited AZO/p-Si, respectively.

In addition, $V_{OC}$ is related directly to the band gap so that enhancement of band gap increases $V_{OC}$. As can be seen in Table 2, the maximum band gap and hence, the maximum amount of $V_{OC}$ are indicated in sample annealed at 500 °C. In an ideal device, $V_{OC}$ is limited by radiative recombination. Because of the unique photoelectrical properties of AZO/p-Si annealed at 500 °C, it is considered as a suitable candidate in solar cell and photodiode applications.

High amount of $n$ in AZO/p-Si diodes maybe due to the presence of surface states or interfacial layers and demonstrates that the transport mechanism is no longer controlled by thermionic emission (Missoum et al. 2016). The forward bias log $I$ vs-log $V$ plot reveals the dominate mechanisms of transport charge in AZO/p-Si diodes along with a power law treatment of current $I \propto V^{m+1}$ where $(m+1)$ changes with the injection level and corresponds to trapping centers distribution (Missoum et al. 2016). As shown in Fig. 10, AZO/p-Si diodes follow three regimes: ohmic, space-charge-limited current (SCLC), and trap filling limit (TFL) (Missoum et al. 2016).

| Table 3 PL peak positions of as-deposited and annealed AZO/p-Si heterojunction |
|-----------------------------|-----------------------------|-----------------------------|
| Sample                      | Peak positions (nm)         | FWHM (nm)                   |
| AZO/Si As-Dep               | 389.55                      | 0.23                        |
|                             | 424.46                      | 72.41                       |
|                             | 481.66                      | 21.10                       |
|                             | 522.67                      | 69.58                       |
| AZO/Si 400 °C               | 379.05                      | 25.21                       |
|                             | 422.31                      | 57.58                       |
|                             | 478.54                      | 37.69                       |
|                             | 521.49                      | 72.36                       |
| AZO/Si 500 °C               | 307.85                      | 1.59                        |
|                             | 414.33                      | 67.64                       |
|                             | 488.86                      | 43.84                       |
|                             | 510.8                       | 121.87                      |
| AZO/Si 600 °C               | 372.7                       | 113.92                      |
|                             | 415.12                      | 64.94                       |
|                             | 439.54                      | 652.71                      |
|                             | 491.05                      | 79.49                       |
In low range of voltage, for as-deposited and annealed AZO/p-Si at 400 °C in region 1 (0.05 < log V < 0.08), annealed AZO/p-Si at 500 °C in (0.2 < log V < 0.3), and annealed AZO/p-Si at 600 °C (0.1 < log V < 0.15), an ohmic mechanism controlled the increase of current with voltage. This is because comparing to background thermal carrier density, amount of perfused effective carrier density is lower (Lee et al. 2004) and it is associated with trapping centers distribution (Lee et al. 2004). As the intensity and density of the trap distribution centers for different annealing temperatures varied in PL analysis, the ohmic region range was also different. In the intermediate range of voltages, for as-deposited and annealed AZO/p-Si at 400 °C (0.08 < log V < 0.1), annealed AZO/p-Si at 500 °C (0.3 < log V < 0.4), and annealed AZO/p-Si at 600 °C (0.15 < log V < 0.1) in region 2, SCLC is the dominate mechanism and the role of voltages is more than region 1. Moreover, comparing to thermal-generated free charge carrier density, the density of perfused free charge is much higher (Singh et al. 2011) which increases the current. In region 3 with high voltages, for as-deposited and annealed AZO/p-Si at 400 °C (0.1 < log V < 1), annealed AZO/p-Si at 500 °C (0.4 < log V < 1), and annealed AZO/p-Si at 600 °C (0.2 < log V < 1), the
exponential increase of current is observed. Here, trap filling limit controlled the mechanism. Injected electrons fills the deep traps until all trap sites become fully occupied. Interestingly, comparing different regions of voltage for all samples shows that they change with almost the same slope and hence, the behavior of their occupied and trap sites are the same.

4 Conclusion

The effect of annealing process on structural, electrical, and optical properties of n-AZO/p-Si heterojunction was investigated in the present study. The fabrication of n-AZO/p-Si heterojunction was carried out by the deposition of AZO thin films on p-Si substrate with magnetron sputtering technique. The XRD results indicated the amorphous structure of as deposited and annealed AZO thin films. FESEM images demonstrated that increasing annealing temperature up to 500 °C increased average diameter because of agglomeration so that the distribution of particles was disrupted. AFM images also illustrated that RMS roughness of samples was increased from 0.42 to 1.61 by enhancement of annealing temperature. Also, by increasing temperature to 500 °C, the band gap of thin films varied from 3.2 to 3.9 eV. Moreover, PL spectra showed that the PL centers and intensity were considerably increased by increasing annealing temperature. Good rectifying behavior of AZO/p-Si heterojunction annealed at 500 °C was also confirmed. Ideality factor, saturation current, rectification ratio \( I_F/I_R \), open circuit voltage \( (V_{OC}) \), and short circuit current \( (I_{SC}) \) were the parameters optimized by the I–V measurements. The dominate mechanisms was transport charge in AZO/p-Si diodes. Since the AZO thin film is in amorphous structure, charge transfers are limited and therefore diode structures could not exhibit very good rectification and photovoltaic properties. However, since making amorphous thin films is easier and cheaper, it has been studied and the remarkable result is that by changing the surface structure of amorphous thin films and eliminating or reducing grain boundaries, electrical properties can be improved.

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![Logarithmic I–V plot of as-deposited and annealed AZO/p-Si heterojunction](attachment:fig10.png)
Declarations

Conflicts of Interest The authors report no conflict of interests. The authors alone are responsible for the content and writing of the paper.

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