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Effect of annealing on the properties of transparent conducting Ag doped ZnO thin films prepared by r.f. magnetron sputtering method

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ABSTRACT

Ag doped ZnO thin films were prepared by r.f. magnetron sputtering method and then heat treated at 450 °C in 2 h in different ambients (air, O_2/O_3). Evolution of morphology, optical and electrical properties of the as-prepared thin films upon annealing environments was studied. XRD analysis demonstrated that annealing had resulted in better crystallization as well as higher transparence. Optical absorption showed that the band gap of the films also reduced after heat treatment. The results also showed that conductivity of the films could be tuned from 10^{-3} Ω cm to 10^{+5} Ω cm by an appropriate annealing process.

1. Introduction

Zinc oxide as a direct band gap semiconductor of wide band gap (3.37 eV at room temperature [1]) similiar to GaN [2] offers various practical applications in different fields such as: cosmetics [3], solar cells [4], gas sensors [5], photo-detectors [6], light emitting diodes [7], photocatalyst [8–10], etc since it is composed of Earth-abundant elements and is safe to humans in contrast to GaN. However, high resistivity of pure ZnO limits its potential in optoelectronics, where high conductivity and high transparency are preferred in many device applications. Enhancement of conductivity of ZnO thin films can be achieved conveniently by doping process. A great effort of material scientists was contributed to engineering optical and electrical properties of ZnO by doping with various dopants such as: indium, aluminum, copper, etc. [11–13]. Ag doped ZnO (here after denoted as ZnO:Ag) thin films were investigated by only a few groups recently [8,14–17]. The results show that silver is a very potential dopant for ZnO materials to obtain highly conductive and transparent thin films for optoelectronics applications such as: transparent electrodes in solar cells, flat-panel displays.

In this paper, ZnO:Ag thin films were prepared by r.f. magnetron sputtering method. Post annealing was demonstrated as an effective way to control structure, morphology and optical transmittance and electrical properties of ZnO:Ag thin films. The correlation between structure, optical and electrical properties of the samples with synthesis condition was presented.

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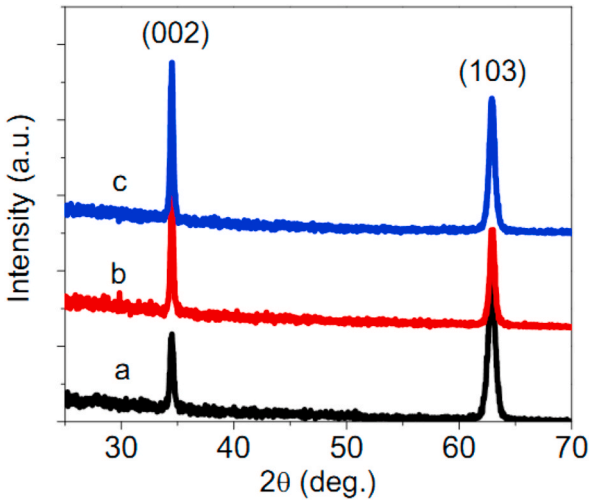

Fig. 1. XRD patterns of undoped ZnO thin films: (a) before annealing, (b) after annealing in air (c) after annealing in O_2/O_3 ambient.

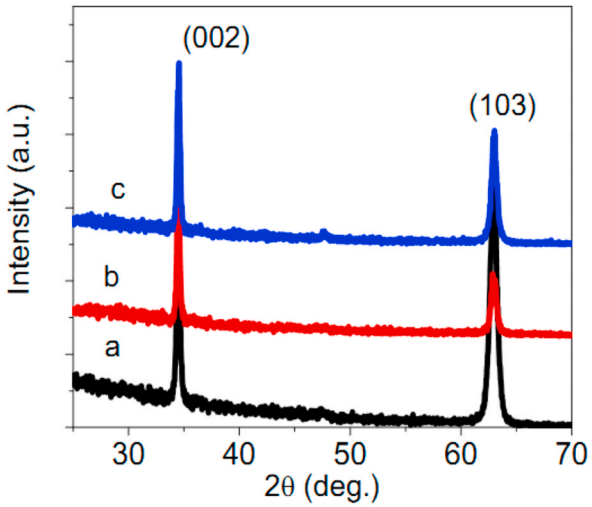

Fig. 2. XRD patterns of ZnO:Ag (1%) thin films: (a) before annealing, (b) after annealing in air (c) after annealing in O_2/O_3 ambient.

2. Experiment

ZnO:Ag thin films with different doping concentration (0 at.%, 1 at.%, 2 at.%, 4 at.%) were deposited on soda-lime glass and silicon substrates by mini sputter ULVAC (Japan). Prior to sputtering, the chamber was pumped down to 10^{-6} Pa. After that, Ar gas was introduced into the chamber to maintain the pressure at 1 Pa. The sputtering power was kept constant at 175 W as suggested by our previous study while sputtering time was set at 20 min. The sputtering process was performed without heating the substrates. The thickness of the films estimated by quartz crystal vibration method was 1.2 μm. Post annealing at 450 °C in 2 h in air and O_2/O_3 ambient was applied to study the changes in properties of the thin films.

Phase structure of the thin films was studied by X-ray diffractometer Bruker D5005 with monochromatic wavelength $\lambda \approx 1.54056$ Å of Cu-K_α radiation in a $\theta/2\theta$ configuration. Surface morphology of the films was observed by atomic force microscope XE-100 Park system. Transmittance and absorbance of the films were collected on Jasco V-750 UV–Vis spectrometer. Electrical properties of the samples were measured with Hall effect Measurement system HMS-3000 using Van Der Pauw technique.

3. Results and discussion

XRD patterns of ZnO:Ag thin films doped with different concentration of Ag: 0%, 1%, 2% and 4% prepared at sputtering power of 175 W without post annealing, with post annealing in air and in O_2/O_3 ambient are shown in Figs. 1–4, respectively.

It can be seen that all the as-prepared thin films without annealing show two distinguished diffraction peaks at 34.4° and 62.9°

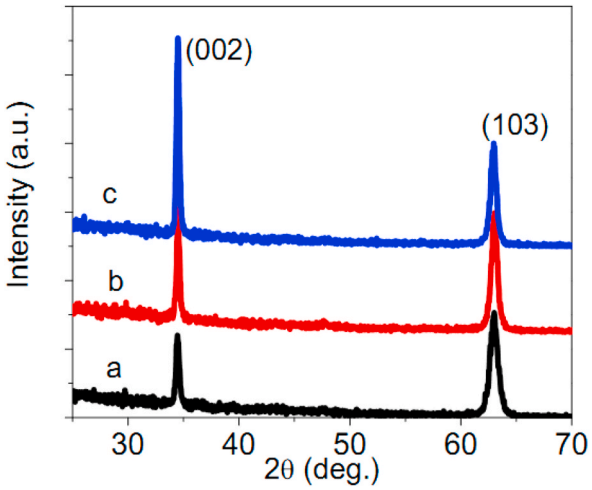

Fig. 3. XRD patterns of ZnO:Ag (2%) thin films: (a) before annealing, (b) after annealing in air (c) after annealing in O_2/O_3 ambient.

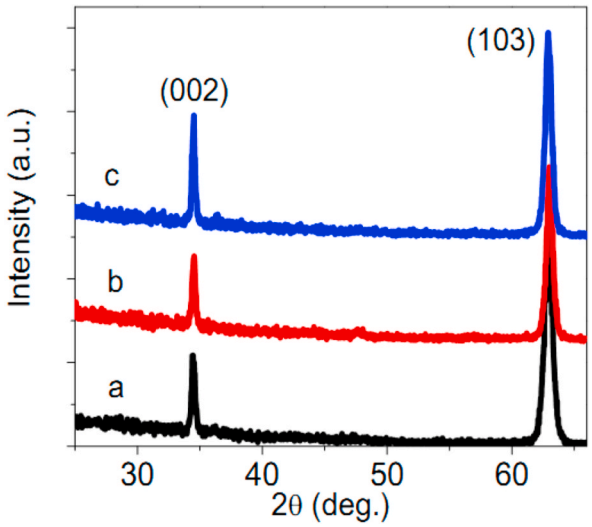

Fig. 4. XRD patterns of ZnO:Ag (4%) thin films: (a) before annealing, (b) after annealing in air, (c) after annealing in O_2/O_3 ambient.

corresponding to the reflection from (002) and (103) planes. The diffraction peaks of all films match well with the JCPDS No. 36–1451 of hexagonal structure of wurtzite. After being annealed, the diffraction peaks grow in intensity and become sharpers, while no other peaks can be observed. The crystal size of the samples was estimated by De Bye Sheerer formula given by:

$$D = \frac{0.9\lambda}{B\cos\theta}$$

where, B is full width at half maximum, θ is the peak position and λ is the monochromatic wavelength of X-ray radiation. The crystalline size increases from 26 nm to 31 nm after annealing process. The results imply that after heat treatment, no phase transition occurred but crystalline quality was enhanced notably.

Another notable feature of the XRD pattern is the increasing of the relative intensity of (002) and (103) peaks induced by heat treatment. After annealing, (002) orientation becomes dominant to (103) orientation except for ZnO sample doped with 4% Ag. Even though the intensity of (002) peak much increases after heat treatment, the (103) orientation is still stronger for this sample.

ZnO thin films synthesized by sputtering methods were often reported to exhibit c axis orientation. (103) texture of ZnO thin films can be only rarely found and only in thin films prepared by wet chemical methods such as sol-gel [18] or electrochemical method [19]. According to the study by Y. Wang [20], (103) orientation was also obtained by sputtering method and likely related to surface structure of the as prepared films due to the movement or diffusion of atoms at the final stage of the sputtering process. During sputtering, the deposited atoms are restricted by the upper layer while at the end of sputtering process, the sputtered atoms have higher freedom so they can move and diffuse more freely similar to ZnO prepared by wet chemical method, leading to the preferred (103)

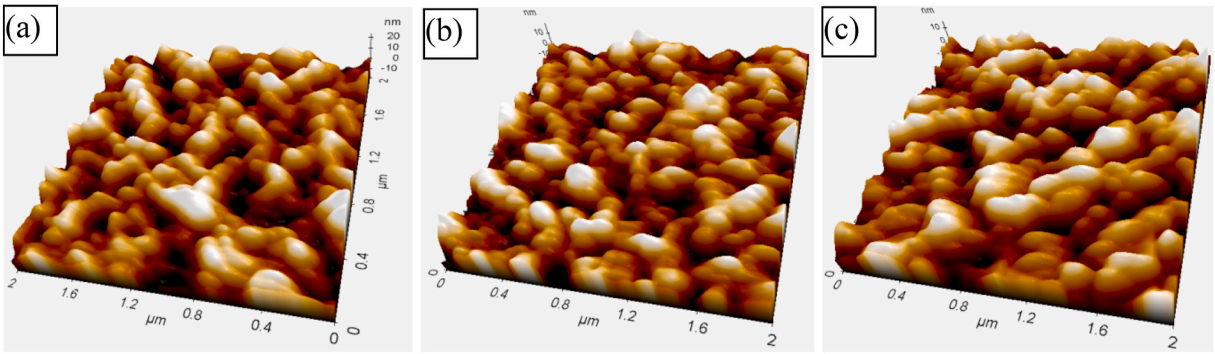

Fig. 5. AFM images of ZnO: Ag (1 at.%) thin films a) before annealing, after annealing (b): in air, c) in O_2/O_3 ambient.

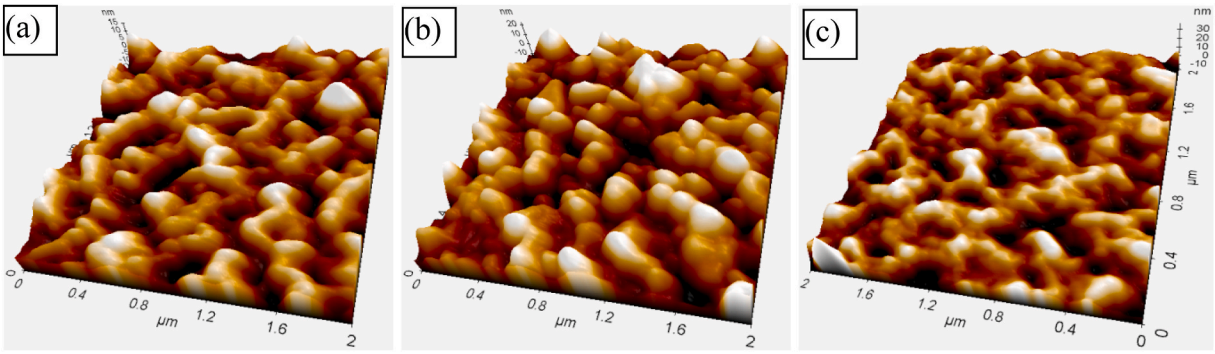

Fig. 6. AFM images of ZnO:Ag (2 at.%) thin films a) before annealing, after annealing (b): in air, c) in O_2/O_3 ambient.

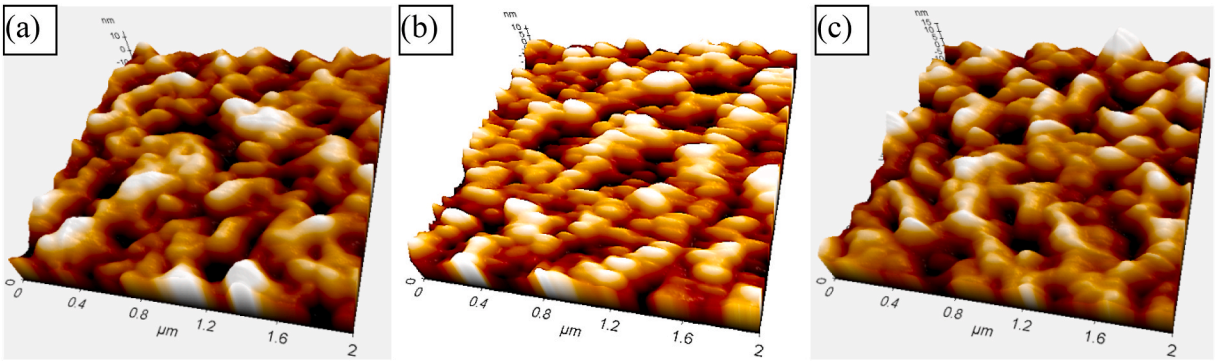

Fig. 7. AFM images of ZnO: Ag (4 at.%) thin films a) before annealing, after annealing (b): air, c) in O_2/O_3 ambient.

orientation of the thin films.

Another explanation for the growth of ZnO thin film along (103) direction is the reduction of surface energy of (103) plane due to dopants. In pure ZnO material, the surface energy of (002) plane is the lowest and results in the growth along c axis. However, various reports showed that doping can change surface energy and results in the growth of thin films or nanostructures with preferential orientation different from (002). For examples, Al [21], In Refs. [22,23] or Ga [24] doping was report to result in the alteration of surface energy of different facets in ZnO crystal, which in turn let the growth of other planes be possible.

In this study, the change in surface energy is likely more suitable explanation. First, I_{103}/I_{002} XRD intensity ratio is different for as-deposited thin films of different doping concentration even though they are prepared exactly at the same condition. Second, it can be seen that the higher doping concentration is, the more clearly (103) orientation exhibits. The last evidence for this argument is the transformation of XRD pattern of the thin films after calcination. An intensity reduction of the reflection from (103) plane was induced by annealing process.

As can be seen from the XRD patterns, (002) peaks of the ZnO:Ag thin films shifted systematically to the higher angle position after

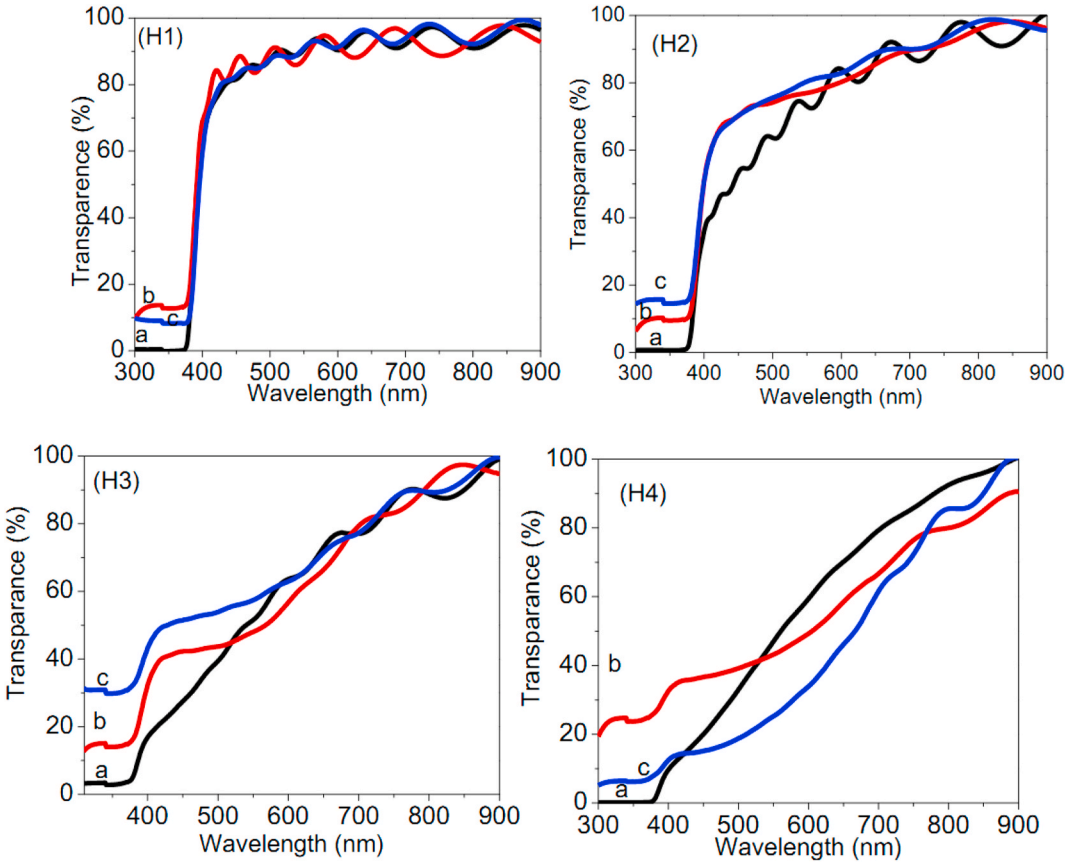

Fig. 8. Transmission spectra of ZnO (H1) and ZnO:Ag (H2: 1%, H3: 2%, H4: 4%) before annealing (a) and after annealing in air (b), in O_2/O_3 ambient (c).

Table 1
Band gap of ZnO and Ag doped ZnO thin films (1%, 2%, and 4%) before and after annealing.

E_g (eV) Sample Annealing condition	ZnO	ZnO:Ag (1%)	ZnO:Ag (2%)	ZnO:Ag (4%)
Without annealing	3.22	3.24	3.13	3.06
Annealing in air	3.18	3.15	3.06	2.84
Annealing in O_2/O_3 ambient	3.17	3.13	3.00	2.84

annealing. The difference in ion radius of Ag^+ (1.22 Å) and Zn^{2+} (0.72 Å) [25] limits the number of Zn atoms substituted by Ag and Ag at interstitial positions might be favorable. The peaks shift indicated the reduced stress of the thin films after heat treatment, which can be attributed to Ag interstitial defect formation. It is reasonable to believe that after calcination, a number of Ag atoms would be removed from substitution positions and results in interstitial defects. This also explains for the variation of I_{003} after heat treatment. As Ag atoms are removed from doping sites, the energy surface of (103) gradually increases and as a result, the preferential orientation turns back to (002). We understand that for ZnO thin films doped with Ag 4 at.%, even though a number of Ag atoms move from substitution sites to interstitial site after annealing, the number of atoms remain as doping atoms is still significant, and hence the (103) preferred orientation is still maintained after annealing.

AFM images of the samples are shown in Fig. 5, Fig. 6 and Fig. 7. The AFM measurements show that annealing process helps the particles become denser and more uniform and the root mean square roughness is greatly reduced. The root mean square roughness of unannealed samples is about 4.6 nm while the roughness decreases to about 3.5 nm after heat treatment.

Heat treatment also exhibits clear effects on the optical properties of the films. We believe that annealing in rich, and active oxygen ambient can effectively release the strain of ZnO thin films to achieve low surface energy, reduce the dislocation of ZnO lattice, as well as strengthen the migration of surface atoms. Such strain release was evidenced by XRD diffraction data as discussed. These processes are beneficial for the growth of high quality films with smoother surface. The transparence of thin films (Fig. 8) with doping concentration of 1% and 2% becomes higher after annealing. Higher transparency might result from the higher smoothness of the films after annealing, which reduce the reflection from the surface of the films. ZnO sample doped with 4 at.% of Ag shows low transparency

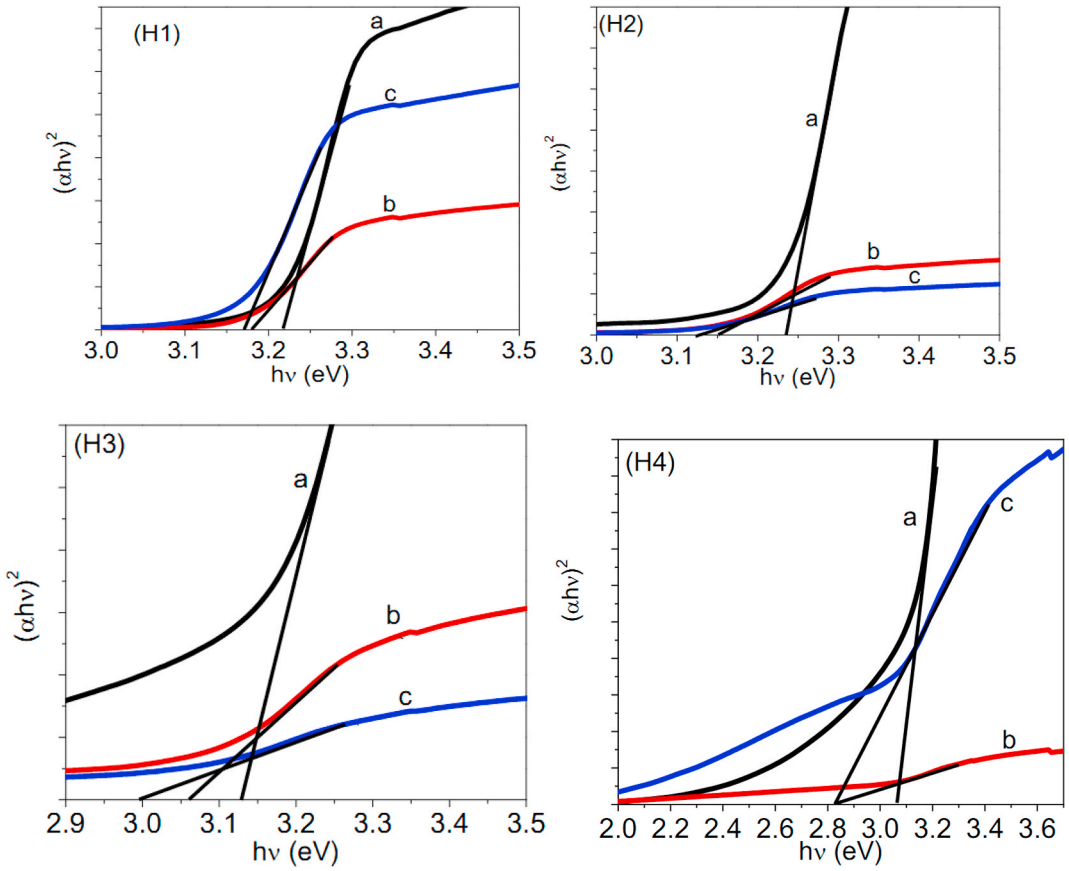

Fig. 9. Tauc plots of ZnO thin films (H1) and Ag doped ZnO with different concentration (H2: 1%, H3: 2%, H4: 4%) before and after annealing process (a: without annealing, b: annealed in air, c: annealing in O_2/O_3 ambient).

Table 2
Hall data of the as-prepared ZnO:Ag thin films with different doping concentration (1%, 2%, 4%).

Sputtering power	Sample	Carrier concentration (x10^{20} cm^{-3})	Mobility (cm^2/V.s)	Resistivity (10^{-3} Ω cm)
175 W	ZnO:Ag (1%)	1.9	18.9	3.1
	ZnO:Ag (2%)	4.9	13.7	1.8
	ZnO:Ag (4%)	0.2	5.5	59.7

after annealing process, which might result from the formation of Ag island after annealing. Such Ag island enhanced reflection of light and as a result the transparency will be reduced.

It can be seen that band gap of the doped thin film is smaller than that of the undoped one [1,26] and band gap decreases with increasing doping concentration correspondingly (Table 1). We believed that dopant had resulted in the decrease of band gap of the films. The results are in agreement with report of Tarwal et al. [27]. The band gaps of the films were also reduced by annealing process as shown in Fig. 9. The results also showed that band gap is further narrowed when annealed in O_2/O_3 mixture. Several possible reasons might contribute to reduce bandgap of the thin films for example: the growth of crystal size induced by annealing or stress release due to oxygen vacancy annihilation during heat treatment in oxygen rich ambient. However further study should be paid to clarify the nature of bandgap narrowing after annealing.

The quality of a tranparent conducting film can be evaluated by figure of merit, defined by Haacke equation [28]:

$$\Phi_{TC} = \frac{T^{10}}{R_{sh}}$$

where, T is the transmistance and R_{sh} is the sheet resistance of the thin film. The figure of merits Φ_{TC} of the as-deposited ZnO:Ag thin films suggest that they are potential for TCO applications. The maximum Φ_{TC} of 3.8×10^{-3} Ω^{-1} was achieved for ZnO doped with Ag 1 at.% thin film. This figure of merit is potential when compared with that of some other inexpensive transparent conducting oxides replacing the TCO-industry standard ITO [29,30].

Hall measurements (Table 2) show that after annealing the films switch to the non-conductive state. Heat treatment somehow alters the intrinsic properties of the films. One possible explanation to that transformation is the motion of Ag^{+} ion from substitutional sites to interstitial sites as suggested by XRD results. The experimental data clearly indicates that post annealing process is an effective way to control the doping of Ag into ZnO and tune the resistivity of ZnO:Ag thin films in a wide range from 10^{-3} Ω cm to 10^{5} Ω cm for various applications in optoelectronics devices.

4. Conclusion

Ag doped ZnO thin films were successfully deposited on glass substrate by r.f. magnetron sputtering. The study showed that annealing ambient is critical to control structure, optical and electrical properties of the films. Rich and active oxygen ambient is beneficial as it helps to improve crystallinity, smoothness and transparency as demonstrated with XRD, AFM and transmission spectroscopy. Conduction state of the films can be actively controlled by appropriate annealing process with the resistivity in the range from 10^{-3} Ω cm to 10^{5} Ω cm which is important for applications in optoelectronics device fabrication.

Author's contribution statement

Nguyen Hai Pham and Viet Tuyen Nguyen conceived of the presented idea. Thi Ngoc Anh Tran and Thi Ha Tran prepared the sample under supervision of Nguyen Hai Pham. Thi Ngoc Anh Tran, Thi Ha Tran, Van Thanh Pham, Van Tan Tran carried out experiment. Thi Ha Tran wrote the manuscript. Quang Hoa Nguyen, Cong Doanh Sai, Nguyen Hai Pham, Thanh Cong Bach, Thi Ha Tran, Thi Ngoc Anh Tran, Quoc Khoa Doan, Van Thanh Pham, Van Tan Tran, Thanh Binh Nguyen contributed to the interpretation of the results. Nguyen Hai Pham and Viet Tuyen Nguyen proofread the manuscript. All authors discussed the results and contributed to the final manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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