



Comparative studies on the properties of magnetron sputtered transparent conductive oxide thin films for the application in solar cell

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Abstract

Transparent conductive ZnO:Al and ZnO:Ga ultrathin films have been developed on glass substrate at room temperature by non-reactive RF magnetron sputtering using sintered ceramic disc of ZnO:Al target (ZnO 98%, Al 2%), and ZnO:Ga target (ZnO 97%, Ga 3%). The thin films (90–250 nm) of ZnO:Al and ZnO:Ga show low resistivity of 8.6×10^{-5} and 4.5×10^{-4} Ω cm, respectively. The ZnO:Al thin film exhibits highest transparency of 92%, haze factor of 54% in the visible region and electrical mobility of 12 $\text{cm}^2/\text{V s}$. Similar kinds of results (highest electrical mobility is 10 $\text{cm}^2/\text{V s}$, haze factor varies from 28 to 44%) are observed for ZnO:Ga thin films. The moderate improvement on thickness-dependent electrical mobility for very thin ZnO:Ga and ZnO:Al films are due to improved crystallinity, increased crystallite sizes but negative effect comes from grain boundary scattering and higher surface roughness. X-ray diffraction spectra reveal polycrystalline nature of ZnO:Ga and ZnO:Al thin films with grain size 22 and 19 nm, respectively. Variation of thickness and composition-dependent structural, morphological and optical haze properties of Al- and Ga-doped ZnO thin films have been compared with that of conventional ITO and $\text{SnO}_2:\text{F}$ thin films.

1 Introduction

Transparent conductive doped ZnO thin films are mostly promising materials for many emerging applications like UV photodetectors, flat-panel displays, gas sensors, thin film transistors, piezoelectric material-based MEMS, light-emitting devices, and thin films solar cells due to its unique materials properties such as low sheet resistance, high optical band gap, unique opto-electronic properties and other interesting features like non-toxicity, ease of availability, and high adhesion to substrate materials. [1–9]. The most popular commercially available transparent conducting oxides are indium tin oxide (ITO) and fluorine-doped tin oxide ($\text{SnO}_2:\text{F}$) thin films. The problem regarding ITO films is that the indium contained in ITO, is a rare metal facing the

risk of depletion and typically found in limited geographical areas like China. There is a concern that indium will soon be on the strategic resource lists of every country in the world and alternate replacement is very much essential. Demerits of ITO and $\text{SnO}_2:\text{F}$ films for solar cell applications have been reported by Das et al. [10]. Doped ZnO thin films especially aluminum-doped zinc oxide (ZnO:Al) (AZO) and gallium-doped zinc oxide (ZnO:Ga) (GZO) thin films can overcome the above-mentioned problems and fulfill the demands of application concerned in the above-mentioned emerging field of technology. ZnO thin films are grown by various deposition techniques such as spray pyrolysis [10], pulsed laser deposition, sol–gel process, electron beam evaporation, molecular beam epitaxy, DC and RF magnetron sputtering, and chemical vapor deposition [11–21].

However, most of the deposition techniques require intentional heating at moderate temperatures to obtain good electrical properties. Non-reactive radio frequency RF magnetron sputtering is one of the most appropriate techniques for the preparation of such ZnO-based TCO materials. RF magnetron sputtering plays an important role in the deposition of ion damage-free uniform film with high deposition rate and good adhesion. Hung-Wei Wu et al. studied ZnO:Al thin film of thickness 100 nm deposited on glass substrate by RF sputtering at the power of 250 W with Ar flow of 40

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sccm and achieved lowest resistivity of $6.11 \times 10^{-4} \Omega \text{ cm}$ and highest transmittance of 92.8% [22]. En-Gang Fua et al. prepared ZnO:Al films of thickness 143 nm with resistivity of $4.6 \times 10^{-4} \Omega \text{ cm}$ and sheet resistance of $32.16 \Omega/\square$ [23]. According to their report, they studied only the electrical and the optical properties of the film.

Agura et al. [24] developed ZnO:Al (AZO) films on glass substrate by PLD using ArF excimer laser and reported the lowest resistivity and average visible transmittance of $8.54 \times 10^{-5} \Omega \text{ cm}$ and 88%, respectively, for 280 nm thick films. Thickness-dependent structural, electrical and optical properties of AZO films deposited using PLD techniques were reported by Dong et al. [25]. El Hamali et al. [26] prepared 180 nm thick AZO films at room temperature on fused silica substrates by magnetron sputtering technique and reported lowest resistivity of $5 \times 10^{-4} \Omega \text{ cm}$ with visible transmittance of $\sim 85\%$. Similar kinds of work on RF magnetron sputtered ZnO thin films were studied by Nihan Akin et al. [27] and reported improved electrical resistivity with $2.78 \times 10^{-5} \Omega \text{ cm}$, carrier densities over $3.35 \times 10^{21} \text{ cm}^{-3}$, and maximum Hall mobility $11.13 \text{ cm}^2/\text{V s}$. with poor optical transmittance of about 75%. Akio Suzuki et al. [28] deposited 150–200 nm thick ZnO:Al thin films at room temperature on glass substrates by a pulsed laser deposition and lowest electrical resistivity and maximum visible optical transmittance of $5.62 \times 10^{-4} \Omega \text{ cm}$ and 90%, respectively. IZO:Ga films are low resistivity ($1.5 \times 10^{-3} \Omega \text{ cm}$) and low sheet resistance ($20 \Omega/\square$) with average optical transmittance 86%, large Haze factor ($> 70\%$) and high figure of merit has been reported by Das et al. [29]. But, there is no report regarding details of thickness dependence on haze factor analysis along with the optimized electrical properties of magnetron sputtered doped ZnO thin film.

To establish doped ZnO thin film as alternative transparent conductor for replacement of commercially available transparent conducting oxide (TCO), a detailed study on optical transmittance, haze factor variation, and surface texture analysis along with its electrical properties is essential. Tuning of surface texture to achieve optimum haze factor, optimum figure of merit (FOM), with very low electrical sheet resistance of non-reactive magnetron sputtered doped ZnO-based TCO films with the variation of film thickness for opto-electronic (solar photovoltaic, display technology, etc.) applications has not yet been reported. Thickness-dependent material properties of Ga-doped ZnO thin films with $10^{-3} \Omega \text{ cm}$ resistivity developed by Sol–Gel technique have already been reported in *Journal of Materials Science: Materials in Electronics*, vol. 28, issue 6, (2017) 5021. Preparation process of ZnO:Ga film is not magnetron sputtering and they have not reported about haze factor as well as surface texture. In our work, ZnO:Al thin film with $10^{-5} \Omega \text{ cm}$ and ZnO:Ga thin film with $10^{-4} \Omega \text{ cm}$ resistivity have been developed at room temperature by RF magnetron sputtering.

Details of haze factors and figure of merits of ZnO:Ga and ZnO:Al films with controlled surface texture have also been reported.

In this study, we have investigated the effect of thickness variation on electrical, optical (haze), structural, and morphological properties of room temperature-deposited RF magnetron sputtered ZnO:Al, ZnO:Ga films on a glass substrate. The results have been compared to that of conventional commercially available TCOs, i.e., ITO and SnO_2 :F thin films.

2 Experiment

ZnO:Al and ZnO:Ga thin films were deposited on glass substrate at substrate temperature (T_s) 300 K (room temperature) by dual-target RF (13.56 MHz frequency) magnetron sputtering system (Hind High Vacuum Co. (P) Ltd.) under non-reactive environment with Argon ambient. Here, 99.99% pure sintered ceramic disc were used as sputtering targets (2 in. in diameter and 5 mm in thickness) and target specifications are ZnO:Ga (3 wt%), ZnO:Al (2 wt% Al), ITO [indium oxide (90%) and tin oxide (10%) by weight], respectively.

Doped ZnO and ITO thin films were prepared at $T_s = 300 \text{ K}$ using Ar as sputtering gas with a constant substrate rotation at 100 W RF power. Ar flow was maintained at 40 sccm throughout the deposition process. The target was placed at top of the chamber and the substrate at the bottom. Pre-sputtering of all the targets was carried out in pure argon plasma atmosphere for about 15 min to remove the surface oxide layer of the target and the glass substrate was also plasma cleaned before thin film deposition.

Film thickness of TCO thin film samples was estimated by in situ quartz crystal monitor attached with magnetron sputtering system. The fine control of film thickness and uniformity are made by controlling the rotational speed of substrate holder as well as time of film deposition. The electrical properties of all TCO thin films were studied by 4-probe van der Pauw technique attached with Hall measurement (Ecopia-HMS-3000) set-up. Optical transmittance (diffuse, total and absorbance) of thin films was measured using microprocessor-controlled double beam UV–VIS (Perkin Elmer Lambda-35) spectrophotometer. Details of the measurement mechanism for diffused and haze have been discussed elsewhere [10]. The structural characterization of ZnO:Al and ZnO:Ga thin films was carried out by X-ray diffraction (XRD) (Philips PW 1710 diffractometer, Cu K α radiation, $\lambda = 1.54178 \text{ \AA}$, 2θ scan mode). The surface morphology and topography of Al- and Ga-doped ZnO thin films were performed by scanning electron microscopy (FESEM, Inspect F-50, FEI), and atomic force microscopy (AFM) (multimode 8, Bruker), respectively.

3 Results and discussion

ZnO thin films were deposited with 100 W RF power, 10 mbar chamber pressure, at room temperature with controlled substrate rotation under non-reactive environment. The process chamber was evacuated up to a base pressure of 5.6×10^{-6} mbar. Target to substrate distance was kept at 6 cm for all experiments. ZnO:Al and ZnO:Ga films show promising electrical properties with $\rho = 8.6 \times 10^{-5}$ Ω cm, $R_{sh} = 5$ Ω/\square , and $\rho = 4.5 \times 10^{-4}$ Ω cm with $R_{sh} = 26$ Ω/\square and optical transmission of 92 and 83%, respectively, due to the high carrier density, whereas the mobility is 2–3 times less compared to commercial TCO films. The electrical resistivity and Hall measurements have been carried out at room temperature on doped ZnO thin films. The carrier density and mobility of the thin films were estimated from Hall measurements.

The dependence of the resistivity, Hall mobility and carrier concentration on the thickness of doped ZnO films are shown in Table 1. It can be seen that, the carrier concentration increases from 8.68×10^{20} to 5.31×10^{21} cm^{-3} and from 9.34×10^{20} to 1.38×10^{21} cm^{-3} for ZnO:Al and ZnO:Ga thin film of thickness 90–170 nm, respectively. Hall mobility of doped ZnO thin films monotonically increases with film thickness and it is 12 and 10 $\text{cm}^2/\text{V s}$ for 170 nm thick ZnO:Al and ZnO:Ga thin films, respectively. A similar kind of work related to thickness-dependent electrical and structural properties of RF magnetron sputtered ZnO:Ga films have been reported by Yu et al. [30]. They have shown thickness variation from 100 to 1000 nm and upto 400 nm film thickness carrier density monotonically increases, and then saturates. In our work, thickness-dependent materials' properties have been reported to be upto 250 nm film thickness for magnetron sputtered ZnO:Al and ZnO:Ga films. Carrier mobility in thin film structure generally depends on crystallinity as

well as impurity scattering, grain boundary scattering and surface roughness scattering. For ultrathin film, only nucleation takes place and discontinuous films are produced. But with increase of film thickness, bigger crystallites are formed that in turn improves crystallinity as well as weakens inter-crystallite boundary scattering and increases carrier lifetime. This is the cause for improved mobility for Ga-doped ZnO thin films [31, 32]. Similar explanations have been reported by Kim et al. [33].

The details of the results on the electrical properties and the optical properties of ZnO:Al and ZnO:Ga thin films along with that of commercially available ITO and $\text{SnO}_2:\text{F}$ thin films are presented in Table 1 for direct comparisons. Commercial TCO shows very low sheet resistance (< 5 Ω/\square) due to both high carrier density ($\sim 10^{20}$ cm^{-3}) and mobility. Uncertainty in thickness is ± 5 and ± 3 nm for ZnO:Al and ZnO:Ga films as, respectively, shown in Table 1.

Columnar growth of grains is commonly observed in magnetron sputtered doped polycrystalline ZnO thin films [34–36]. Both ZnO:Al and ZnO:Ga films show hexagonal wurtzite structure. When the deposition begins, there are many nucleation centers on the substrate and small crystallites are produced. During nucleation stage, i.e., for ultrathin doped ZnO film, Zn atoms bind to the O-terminated surface, forms ZnO cluster and diffuses across the surface [37, 38]. Due to frequent impact of sputtered atoms with substrate surface, more kinetic energy is transferred to the substrate surface in sputter deposition, hence growth is generally more complete, and the ZnO films become denser and almost free from stacking faults. Ultrathin ZnO films are discontinuous but with increase of film thickness ZnO films become more uniform and homogeneous, so that carriers may get continuous network in doped ZnO films. Combined effect of higher carrier density as well as improved mobility resulted in lowering the sheet resistance and film resistivity of polycrystalline ZnO:Al and ZnO:Ga thin films [39, 40].

Table 1 Variation of electrical and optical properties with TCO film thickness

Thickness (nm)	Resistivity (Ω cm)	Sheet resistance (Ω/\square)	Mobility (μ) ($\text{cm}^2/\text{V s}$)	Carrier concentration (cm^{-3})	Figure of merit (FOM)	% transmittances	
ZnO:Al	90 \pm 5	4.3×10^{-4}	47	10	8.68×10^{20}	3.89×10^{17}	89
	110 \pm 5	9.8×10^{-5}	9	11.6	8.83×10^{20}	6.33×10^{17}	90
	170 \pm 5	8.6×10^{-5}	5	12.0	5.31×10^{21}	7.23×10^{18}	92
	250 \pm 5	5.0×10^{-4}	20	11.0	7.10×10^{20}	1.08×10^{18}	90
ZnO:Ga	90 \pm 3	6.2×10^{-4}	68	8.0	9.34×10^{20}	1.16×10^{17}	80
	110 \pm 3	5.9×10^{-4}	53	8.2	9.64×10^{20}	1.71×10^{17}	81
	170 \pm 3	4.5×10^{-4}	26	10.0	1.38×10^{21}	5.96×10^{17}	83
	250 \pm 3	8.7×10^{-4}	35	9.4	7.64×10^{20}	3.92×10^{17}	82
ITO	170	3.4×10^{-4}	2.0	40.84	4.5×10^{20}	1.74×10^{19}	90
SnO₂:F		3.0×10^{-4}	1.8	39	5.34×10^{20}	1.38×10^{19}	87

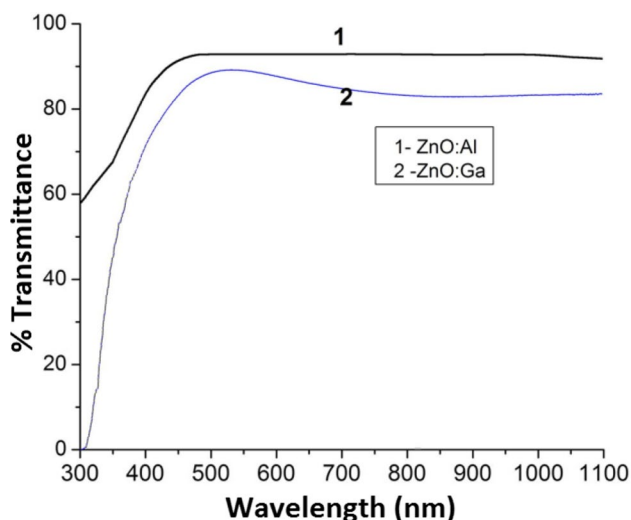


Fig. 1 The optical transmittance spectra of ZnO:Al and ZnO:Ga films

The optical transmittance of the doped ZnO thin films with thickness of 170 nm is shown in Fig. 1. The thin films of ZnO:Al and ZnO:Ga show the highest transmittance of 92 and 84%, respectively, in the visible region. The decrease of transmittance in the near-infrared region results from the increase of reflectance and absorption which is attributed to the plasma resonance of the electron gas in the conduction band. The quality of the transparent conducting films can be judged by the figure of merit (ϕ) calculated from the transmittance (T) and sheet resistance (R_s) data [38] following the equation $\phi = T^{10}/R_s$. The ZnO:Al and ZnO:Ga thin films in our study show the highest FOM of 7.23×10^{18} and 5.96×10^{17} , respectively. The higher values of the figure of merit represent a better performance of the transparent conducting film.

TCO was an alternative of metallic grids used as a front contact to the solar cell, may have the reduced electrical resistance losses. The advantage of TCOs is higher transparency in visible as well as near-infrared region and a high electrical conductivity. Therefore, both the electrical conductivity and the transmittance of TCO film should be as high as possible for application in solar cells.

Figure 2 shows X-ray diffraction (XRD) spectrum of ZnO:Ga thin film (film thickness-170 nm) deposited at room temperature revealing the hexagonal wurtzite structure. GZO sample prepared at room temperature shows diffraction peaks appearing at $2\theta = 31.42^\circ, 34.33^\circ,$ and 37.46° corresponding to (100), (002), and (101) planes, respectively. A prominent (002) peak indicates c-axis orientation of the crystallite that is perpendicular to the substrate plane. The sharp intense peaks confirm the high purity and good crystalline nature of ZnO:Al. The peaks originated along (100), (002), and (103) (shown in Fig. 2 appeared at $2\theta = 31.34^\circ, 34.56^\circ$ and 62.75° , respectively) represent the hexagonal

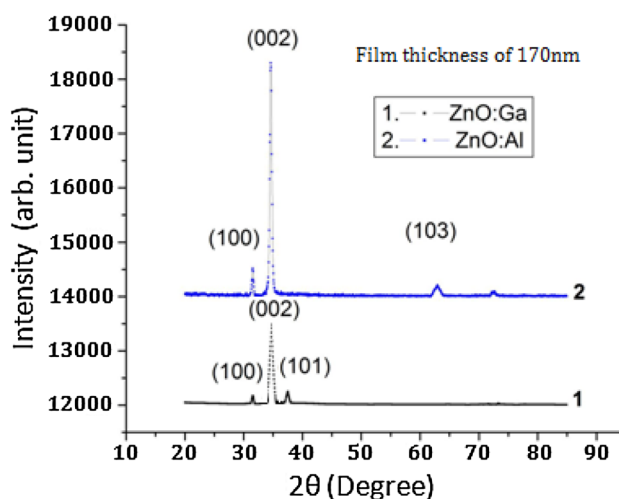


Fig. 2 XRD spectra of ZnO:Al and ZnO:Ga films (film thickness 170 nm)

wurtzite polycrystalline structure of ZnO:Al thin films and strongly preferred c-axis orientation. The crystallite size of TCO thin films was estimated using Scherrer’s formula [41] and for doped ZnO thin films (film thicknesses are 170 and 250 nm respectively), (002) peak is used for grain size calculation (shown in Tables 2, 3).

The spectral haze serves as an indicator for the light scattering properties of the doped ZnO thin films. Doped ZnO thin films shows high diffused transmittance. Variation of haze factor of doped ZnO thin films with different thickness is shown in Fig. 3. Haze values of different TCO films of thickness 170 nm are shown in Fig. 4.

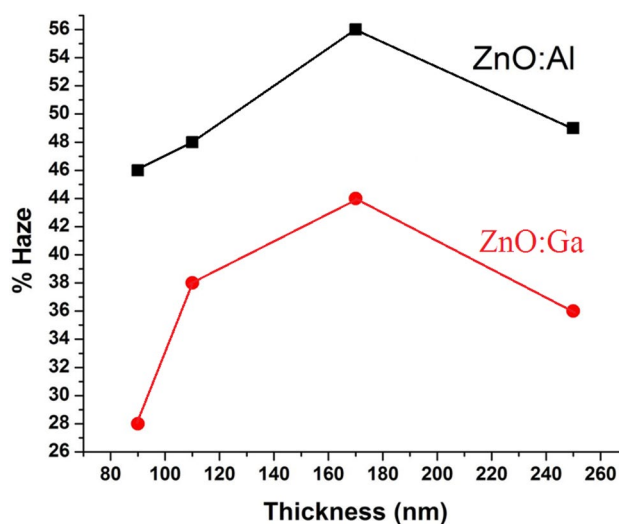


Fig. 3 Variation of haze factor of ZnO: Al and ZnO: Ga thin films of different film thickness

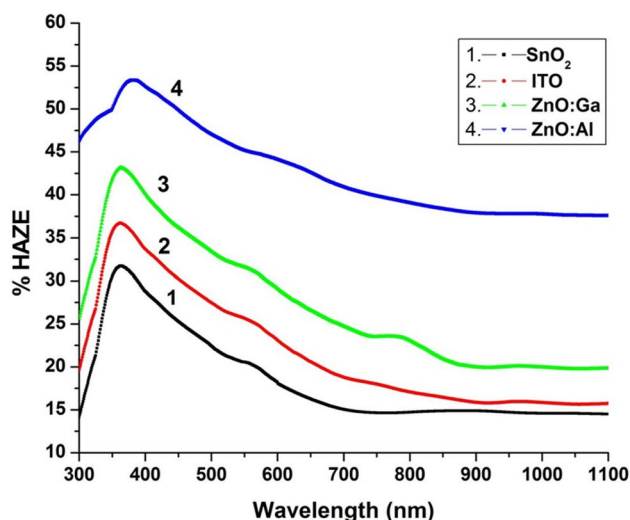


Fig. 4 Haze values of different TCO films

It is observed from graph that doped ZnO thin films have higher haze value.

Haze parameter was derived from the ratio of total transmittance and diffused transmittance. Textured TCO provides light scattering and with increases of diffuse scattering haze factor of the particular TCO surface increases. The haze factor also depends on the surface morphologies of TCO thin films. Controlled texturization of substrate surface by dry etching (ion bombardment) or wet chemical etching (using proper chemicals) can be helpful to make substrate surface rougher and it helps to achieve improved haze factor of TCO films [42].

Therefore, the surface texture of glass substrate and TCO thin films play a vital role to improve the performance of thin film solar cells. A suitable textured surface

is very important to increase the haze ratio and scatter an incident light, particularly the long wavelength light (red and near-infrared). Controlled surface texture of doped ZnO thin films increase the forward light scattering at the TCO/Si interface within solar cell, extending the effective path length within the active silicon layer and subsequent weakly absorbed light trapping inside the absorber material of the solar cell is enhanced.

The detailed morphology and surface roughness of the RF magnetron sputtered ZnO:Al, ZnO:Ga, ITO and SnO₂ thin film were examined by AFM. Figure 5 shows only AFM images of ITO, ZnO:Ga and ZnO:Al thin films. The AFM micrograph shows a high-quality ZnO:Ga thin film with a smooth and crack-free surface.

The surface of the films consists of many granular particles with regular sizes, and it is assumed that the particle/crystallite size increases as the film thickness increases. A similar report was already published in *Applied Surface Science* 245 (2005) 310–315. They reported the resistivity of the film to decrease from 4.9×10^{-3} to $3.1 \times 10^{-4} \Omega \text{ cm}$ when the film thickness increased from 50 to 1000 nm. Film thickness can influence the surface condition and hence materials properties can also vary with the variations of film thickness. Surface roughness, grain size and haze value of the optimized thin films of thickness of 170 nm are shown in Table 2.

Variations of surface roughness, and grain size with film thickness are shown in Table 3. Surface roughness is one of the important properties of the doped ZnO thin films for many opto-electronic applications, because the degree of surface roughness determines the carrier mobility and light scattering, while the smooth structure can reduce the scattering of incident light and increase the transmittance [43]. Similar explanations have been cited by Dimitrov et al. *J Phys Conf Ser* 113, 012044 (2008).

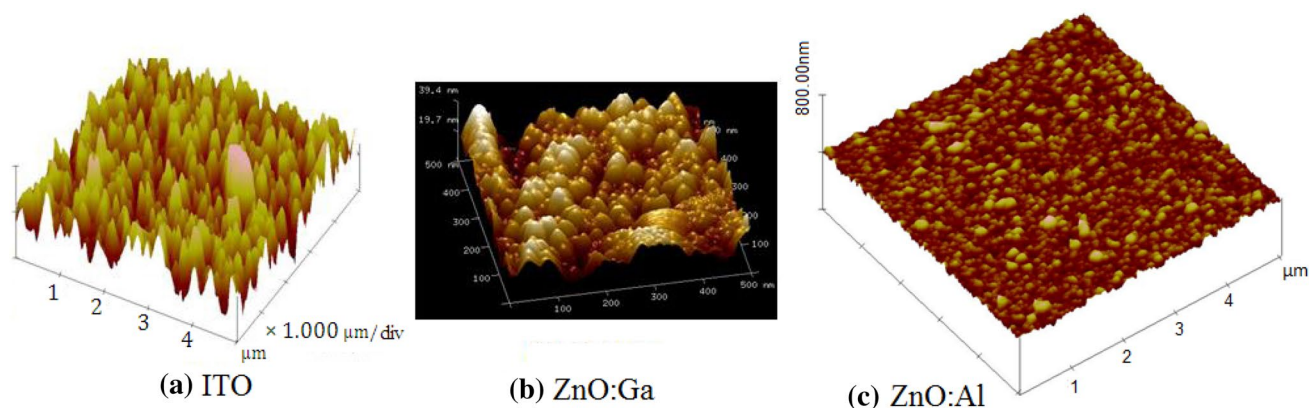


Fig. 5 AFM topography of ITO, ZnO:Ga and ZnO:Al thin films

Table 2 Surface roughness, grain size and haze value of TCO films of 170 nm thickness

Sample (170 nm)	Resistivity (Ω cm)	Sheet resistance(Ω/\square)	Grain size (nm)	Surface roughness (δ_{rms} in nm)	Haze %
ZnO:Al	8.6×10^{-5}	5	19	8.38	54
ZnO:Ga	4.5×10^{-4}	26	22	8.76	44
ITO	3.4×10^{-4}	2	25	19.2	36
SnO ₂ :F	3.0×10^{-4}	1.8	50	36.7	30

Table 3 Surface roughness, grain size with different films thickness

Thickness (nm)	Surface roughness (δ_{rms} in nm)	Grain size (nm)	
ZnO:Al	90 \pm 5	7.84	
	110 \pm 5	9.64	
	170 \pm 5	8.38	19
	250 \pm 5	8.32	19.4
ZnO:Ga	90 \pm 3	8.15	
	110 \pm 3	9.89	
	170 \pm 3	8.76	22
	250 \pm 3	8.68	22.5
ITO	170	19.2	25
SnO ₂ :F	170	36.7	50

4 Conclusions

Transparent conductive ZnO:Al and ZnO:Ga thin films with low resistivity of 8.6×10^{-5} and $4.5 \times 10^{-4} \Omega$ cm, respectively, have been developed by RF magnetron sputtering on glass substrate at room temperature. Both the films are highly transparent over a wide range of visible spectrum with high FOM. High transparency and electrical conductivity are achieved even for very low thickness (170 nm) of ZnO:Al and ZnO:Ga films. Internal texture remains unaltered along *c*-axis orientation with (002) crystallographic direction in both Al- and Ga-doped ZnO thin films. Both AZO and GZO thin films show high haze factor throughout entire solar spectrum and it is very much effective for thin film solar cell. The effect of the film thickness on surface texture as well as to achieve optimized haze factor of room temperature-deposited ZnO-based TCO films on glass substrates have been investigated.

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