

EFFECT OF RAPID THERMAL ANNEALING TREATMENT ON THE ELECTRICAL CONDUCTIVITY OF NANOCRYSTALLINE ZINC OXIDE DOPED WITH ALUMINUM

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Abstract

Spherical nanocrystalline aluminum-doped zinc oxide (nc-AZO) has shown an improvement in conductivity and optical properties under rapid thermal annealing (RTA) treatment. The typical treatment at 500°C for 1 h. on nc-AZO provides low electrical conductivity and low photocurrent, whereas RTA treatment performs better properties with above 85% optical transmittance and higher electrical conductivity. The diameter size of nano-spherical nc-AZO is around at 10-30 nm providing the energy band gap between 3.2 eV and 3.4 eV. Thus, the high electrical and optical qualities of such nc-AZO films can be required for an electron transport layer and transparent contact electrodes for semiconductor devices.

Keywords: AZO; Rapid thermal annealing; Electron transport layer; Conductive improvement; Spherical nanocrystalline zinc oxide

Introduction

Recently, zinc oxide (ZnO) material as a direct energy bandgap and an n-type semiconductor, it high conductivity under UV light is one of the most interesting materials for applied in many potential applications in optoelectronic devices such as the front transparent conductive electrode for silicon

solar cells (Yan *et al.*, 2016), passivation layer for silicon solar cells (Fangsuwannarak *et al.*, 2021), pressure sensor (Samoei and Jayatissa, 2020), transparent conductor for batteries (Ning *et al.*, 2017; Shi *et al.*, 2019), perovskite solar cells for the electron transport layer (ETL) (Zhao *et al.*, 2016;

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Yang *et al.*, 2017). In particular, its wurtzite crystal structure behaves a wide direct energy band gap of 3.20-3.37eV and large excitation binding energy of 60meV at room temperature to be considered as the most promising matrix material for heterojunction photovoltaics. Furthermore, its transparency in the visible light region and adjustable electrical resistivity provides its significant potential which depends on the processing annealing temperature and doping concentration (Krongarrom *et al.*, 2012; Rattanawichai *et al.*, 2018; Fangsuwannarak *et al.*, 2022; Rattanawichai *et al.*, 2022). An additional one of the most crucial advantages of ZnO is the variety of growth methods including sputtering technique (Xia *et al.*, 2017; Daza *et al.*, 2021; Mohamedi *et al.*, 2022), evaporation technique (Constantinescu *et al.*, 2014; Rajesh *et al.*, 2022), spray pyrolysis technique (Nasiri and Rozati, 2018; Mahesh and Kumar, 2020), and sol-gel technique (Efafi *et al.*, 2014; Khan *et al.*, 2022). This further contributes to the intense interest in ZnO for emerging solar cell. However, some of these techniques rely on the high annealing temperature to simultaneously form a nanocrystal structure

Doping in semiconductor material is a very useful means of improving charge separation. The dopants are used for ZnO material such as aluminum (Islam *et al.*, 2019), boron (Bose *et al.*, 2020), gallium (Thambidurai *et al.*, 2014; Ponja *et al.*, 2020), indium (Dev *et al.*, 2020), germanium (Shan *et al.*, 2008) and titanium (Wan *et al.*, 2014) can enhance the optical and electrical properties of ZnO.

The high dielectric constant encourages efficient charge transfer into solar cells. Applying a high dielectric constant material as a cathodic buffer layer is typically possible to form a spontaneous polarization to create an internal electric field. Meanwhile, a mechanism for operating solar cells requires a reduction of the electron recombination, which can contribute to improving the efficiency of power conversion (Lan *et al.*, 2014).

In recent years, rapid thermal annealing (RTA) has become accepted as the mainstream thermal process in the semiconductor manufacturing industry. During the RTA process, the wafer is heated up and cooled down rapidly, thus reducing the diffusion in a solid state of the dopants introduced in the previous manufacturing stages. It leads to a low parasitic resistance (Hao *et al.*, 2015; Rattanawichai *et al.*, 2022). The mechanism for improving optical and electrical stability improvement due to RTA still needs further study.

In this research, The nc-AZO nanostructure was prepared by sol-gel technique with various Al dopants concentrations from 0.0at.%, 0.4at.%,

0.8at.%, 1.0at.%, 1.2at.%, 1.6at.%, and 2.0at% AZO films with a number of layers from 1layer to 5layer were synthesized by spin coating on a quartz substrate. Optical and photocurrent properties of spherical nc-AZO films have been investigated to validate the optimum condition. To accomplish in the ETL function for the emerging solar cell, the RTA condition preparations of the spherical nc-AZO films were concerned at high-temperature effects from annealing to 500°C for 1 hour and RTA at 930°C for 20 seconds. The optimal property of spherical nc-AZO on quartz substrate is closely relevant to its conductivity and its surface morphology features, resulting in high solar cell efficiency. The novelty of this study focuses on the RTA process applied to the spherical nc-AZO multilayers on quartz substrate. This is demonstrated a beneficial influence on the structural, electrical and optical properties, which makes them highly suitable for use as efficient electrodes in third-generation photovoltaic and low-cost semiconductor devices.

In this study, the surface morphology of samples was imaged by using a field emission scanning electron microscope (FESEM), Carl Zeiss machine Auriga, with the magnification 100,000 times. The electrical characteristic (J-V curve) under dark and illumination (1.77mW/cm²) conditions was measured by using a Keithley (Series 2400 Electrometer) with 2-probe Ag contacts. The optical transmittance and reflectance were measured by UV-VIS spectrophotometer (Cary300) in mode integrating sphere with the range of 250-850 nm. The film thickness was measured by an optical profiler (Bruker) and the crystal quality and size of nc-AZO films were verified by an X-ray diffractometer technique (D8 ADVANCE XRD) using Scherrer's equation.

Materials and Methods

Figure 1 showed the preparation schematic diagram of zinc oxide film doped with aluminum (Al) prepared with the sol-gel technique. Substances used in the synthesis of zinc oxide thin film doped with Al, consist of zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$: ZnAC) (CARLO), Isopropyl alcohol ($\text{C}_3\text{H}_8\text{O}$: IPA) (CARLO), aluminum chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) and Ethanamine ($\text{HOCH}_2\text{CH}_2\text{NH}_2$: MEA) (CARLO). Firstly, dissolving ZnAC in IPA at 0.2M concentration and then mix MEA: ZnAC at 0.75M concentration and adding AlCl_3 0-2.0at. % concentration. The solution was stirred at 60°C for 60 minutes, subsequently aging the gel for 24 h. (Salam *et al.*, 2013).

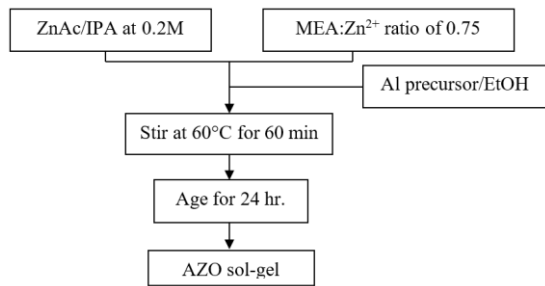


Figure 1. Preparation steps of ZnO:Al sol-gel

After aging ZnO:Al sol-gel for 24 h, the quartz substrate were cleaned by the RCA method (Ratio Corporation of America; RCA). The ZnO:Al films were deposited by a spin coating technique with 2 speed steps at 500rpm for 15 seconds and 2000rpm for 15 seconds. The thin films were dried at 200°C for 30 minutes for 1 layer. And then spin coat again for multilayer. Subsequently, ZnO:Al multilayer thin films were annealed at 500°C (heating rate 1°C/s), maintained at this temperature for 1 h, then cooled down to room temperature at a rate of 1°C/s. To introduce conductive improvement

intentionally, ZnO:Al multilayers/quartz samples were subjected to the RTA process at firing times 930°C for 20 seconds.

Results and Discussion

Figure 2 shows the surface morphology of nc-AZO thin films with various Al dopants concentrations coated on quartz substrates with RTA at 930°C for 20 seconds as observed by FESEM measurement at 100k magnification. Found that, the surface morphology shows small nano spherical size of approximately 10-30nm. Increasing Al dopants concentrations from 0.0at% to 1.2at% to provide similar surface morphology with uniform roughness. Meanwhile, more Al dopants concentrations from 1.6at% to 2.0at% to appear cracking of films increase.

Figure 3 (a) shows the thickness of nc-AZO films with various thicknesses, it is found that nc-AZO film 1layer provides a thickness of approximately 37nm. For increasing the number of layers for 2 layer, 3 layer, 4 layer, and 5 layer to provide thickness as 71 nm, 96 nm, 160 nm, and 249 nm respectively

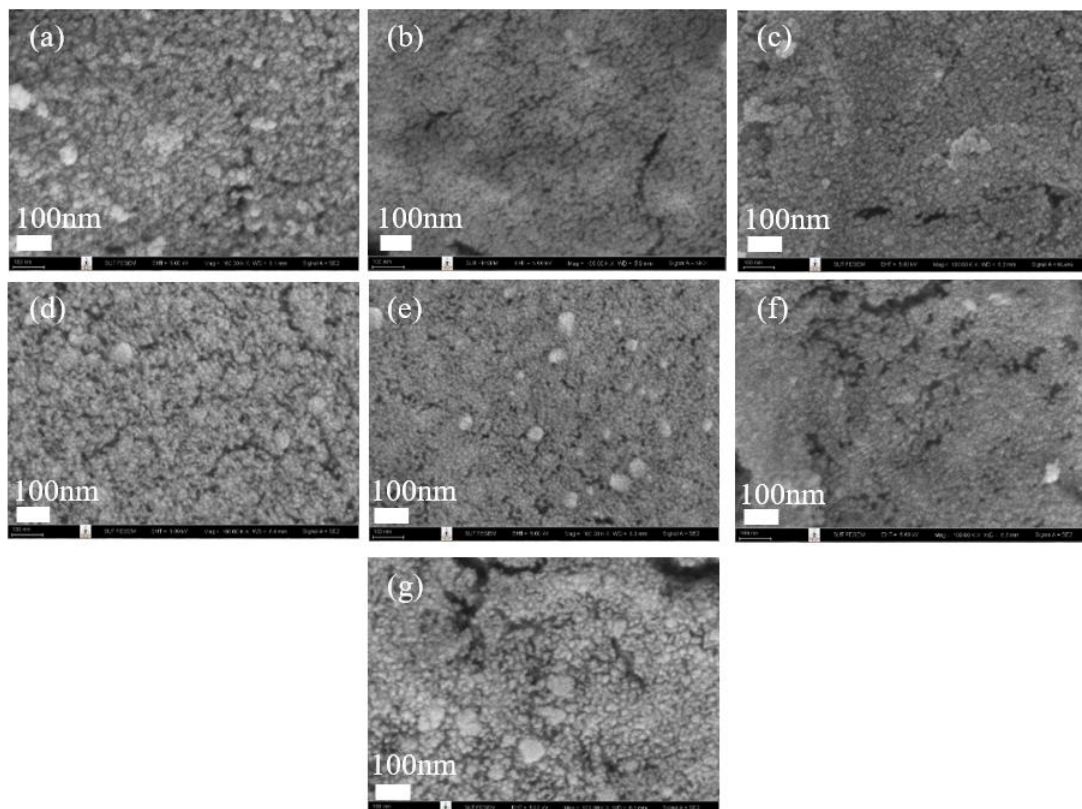


Figure 2. FESEM images with 100k magnifications (a) 0.0at% (b) 0.4at% (c) 0.8at% (d) 1.0at% (e) 1.2at% (f) 1.6at% and (g) 2.0at% of nc-AZO thin films

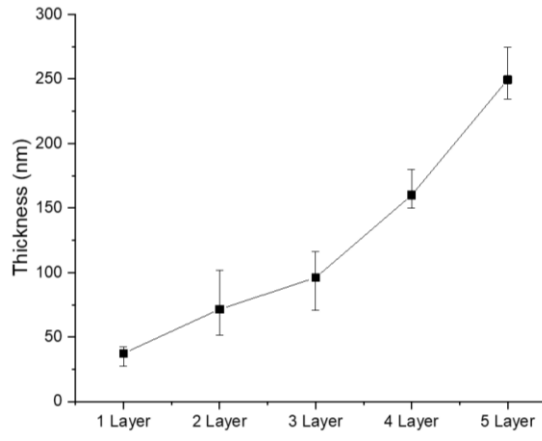


Figure 3. Thickness of nc-AZO films by an optical profiler

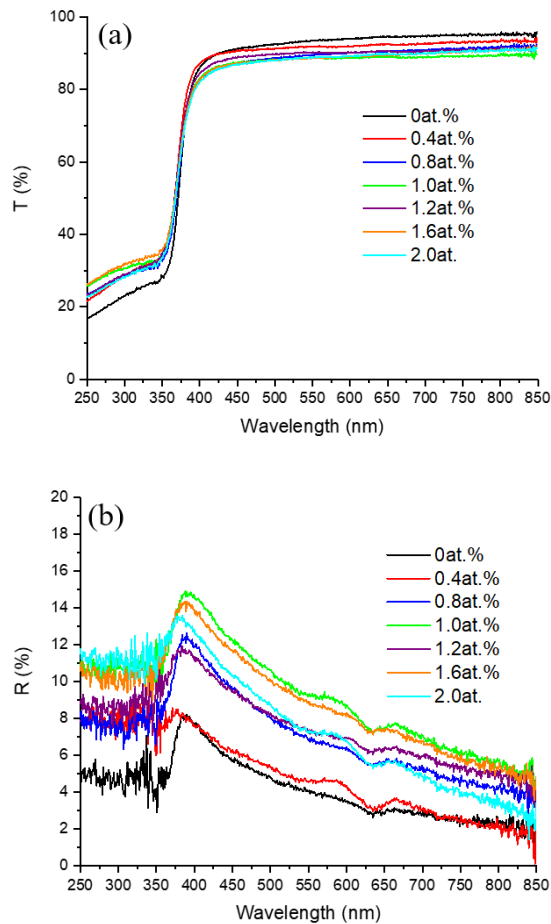


Figure 4. (a) Transmittance spectra (T%) and (b) reflectance spectra (R%) of nc-AZO with various Al dopants concentrations

Figure 4(a) shows the transmittance and (b) reflectance spectra of nc-AZO films in the wavelength range of 250-850 nm with various Al dopants concentrations. In Figure 4(a), it is found that all samples of Al dopants concentrations exhibit transmittance over 85% in the wavelength range of 400-850 nm. In the UV range (250-380 nm), nc-AZO films affect a steep decline in the transmittance spectra.

The nc-AZO films were doped with various Al dopants concentrations to provide the minimized light reflectance of about 10% in the wavelength range 450-850 nm.

The nc-AZO film with RTA-treatment process at 930°C for 20 seconds provided maximized light transmittance and minimized light reflectance perhaps due to the enlarged grain size of the spherical nanoparticles with less grain boundary. Because grain boundary can simultaneously absorb visible light and decrease carrier mobility, enlarged grain size results in the higher visible transmittance (Peng *et al.*, 2016; Choe *et al.*, 2019).

Figure 5. illustrates the reflective index spectrum (n) of nc-AZO on quartz substrate with various Al dopants concentrations. In the visible range, samples were annealed by RTA-treatment at 930°C for 20 seconds. Samples provided a reflective index of about 1.6-2.3. The refractive index will be decreased when increasing the wavelength range. The reflective index of nc-AZO on quartz substrate depends on the Al dopants concentrations. The Al dopants concentrations at 1.0at% and 1.6at% sample to shown higher refractive index than another sample. The high refractive index gradient with temperature was found for films of a nonuniform distribution and gathered in clusters and a high predicted value for thermal diffusivity (Mohammed *et al.*, 2014). However, at 1.0at% sample provided good surface morphology as observed by the FESEM result.

The real dielectric constant ϵ_1 as a function of wavelength is shown in Figure 5(b). For all samples, there are one peak in ϵ_1 near the energy bandgap, which means a strong interaction between photons and electrons at this wavelength (Gadallah and El-Nahass, 2013). The ϵ_1 value in the vicinity of the bandgap wavelength is approximately 3.0-5.0. At 850 nm wavelength, undoped ZnO film and 0.4at% samples provided the dielectric constant of 2.0. while for 0.8at%, 1.0at%, 1.2at%, 1.6at% and 2.0at% samples it is of 3.0. However, at 1.0at% sample provided high dielectric constant in the wavelength range of 500-850 nm and high than undoped ZnO film with good surface feature and

electric property. The imaginary dielectric constant ε_2 of the prepared samples as a function of the wavelength is depicted in Figure 5(c). All sample provides the same value.

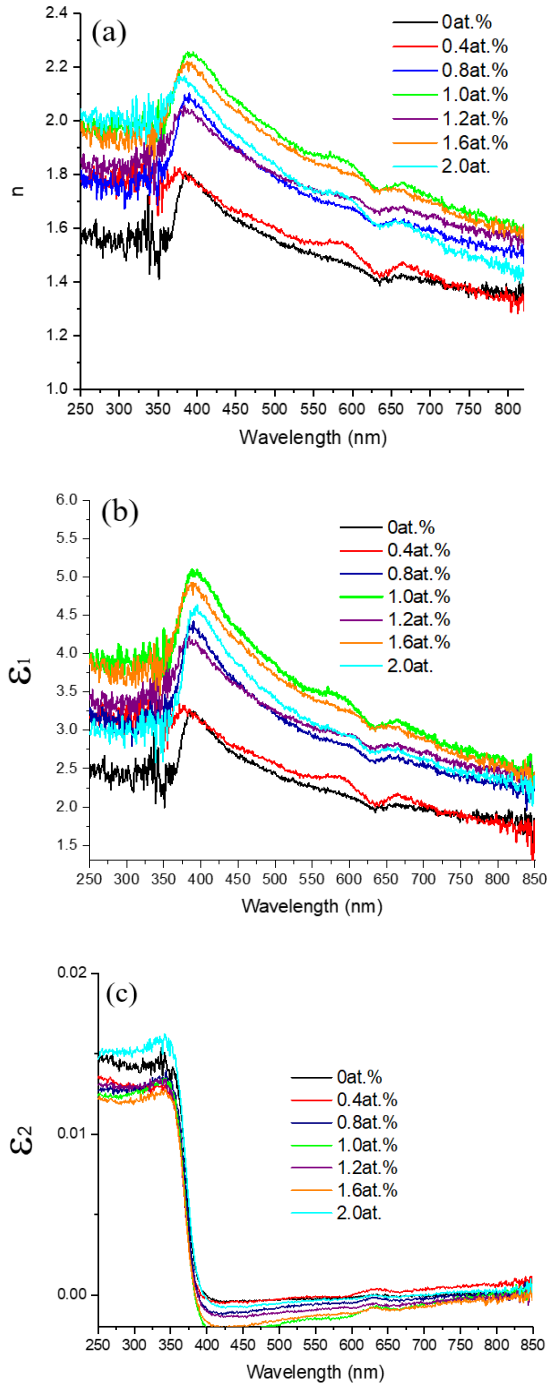


Figure 5. (a) the reflective index spectrum (b) Real part of dielectric constant and (c) Imaginary part of dielectric constant of nc-AZO films

As a result of spherical nc-AZO behaving as a direct bandgap semiconductor, the higher incident photon light than optical band gap (E_g) of spherical nc-AZO film can produce electron-hole pairs without much momentum loss. Thus, E_g implies the minimum energy, which can be absorbed in a semiconductor material and its carriers can be excited to become free carriers. E_g can be estimated by an extrapolation of the linear curve portion as a function of absorption coefficient (α) depending on photon energy ($h\nu$) as shown in the equation (1) (Pankove and Kiewit, 1972).

$$(ah\nu)^2 = C(h\nu - E_g) \quad (1)$$

where C is a constant for a direct transition of photon energy.

The optical absorption coefficient of thin films can be calculated by (Optical Characterization, 2005).

$$\alpha = \frac{1}{t} \ln \left(\frac{\sqrt{(1-R)^4 + 4T^2R^2} - (1-R)^2}{2TR^2} \right) \quad (2)$$

where t is the thickness of the film, T and R are the transmittance and reflectance of the film, respectively.

Figure 6 illustrates the plot of the relationship between $(ah\nu)^2 \times 10^{10} (cm^{-1}eV)^{-2}$ and $h\nu$ for spherical nc-AZO film with various Al dopants concentrations conditions. It is noted that the estimated E_g of undoped ZnO film is of about 3.27eV but after increasing Al dopants concentrations E_g value is shifted into high energy at 3.30eV. The energy band gap shift probably occurs due to the different electron density of the film (Wang *et al.*, 2014; Choe *et al.*, 2019).

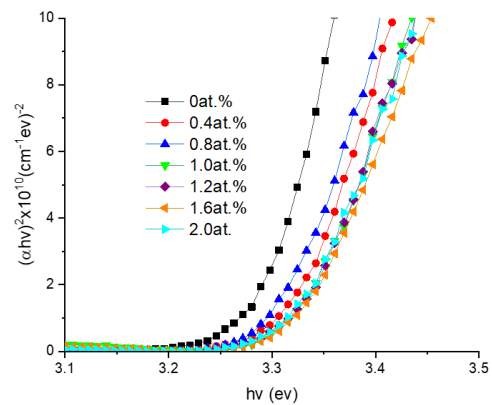


Figure 6. The plot of $(ah\nu)^2 \times 10^{10} (cm^{-1}eV)^{-2}$ in the function of $h\nu$ of nc-AZO films with various Al dopants concentrations

Figure 7 presents the J-V curve in a semi-log scale of spherical nc-AZO films with various Al dopants conditions under dark and illumination tests. It is noted that the films with all preparation conditions measured in an illumination condition show the increasing gain of photocurrent density compared with the dark current. As observed in Figure 7 the undoped ZnO film provided a lower photocurrent of 4-fold, whereas doped ZnO films provided the trend of risen photocurrent density of 830-fold with increasing dopant level.

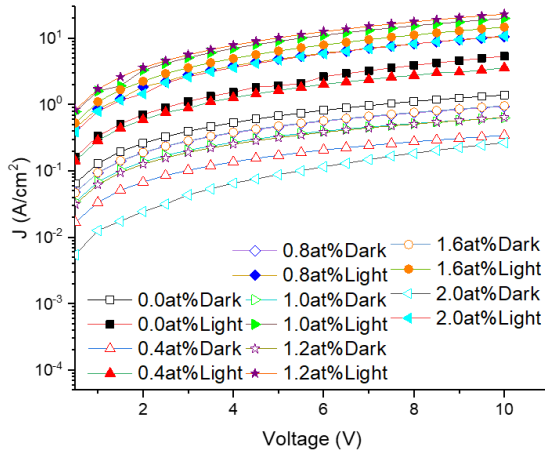


Figure 7. The plot of $(\alpha h\nu)^2 \times 10^{10} (\text{cm}^{-1} \text{eV})^{-2}$ in the function of $h\nu$ of nc-AZO films with various Al dopants concentrations

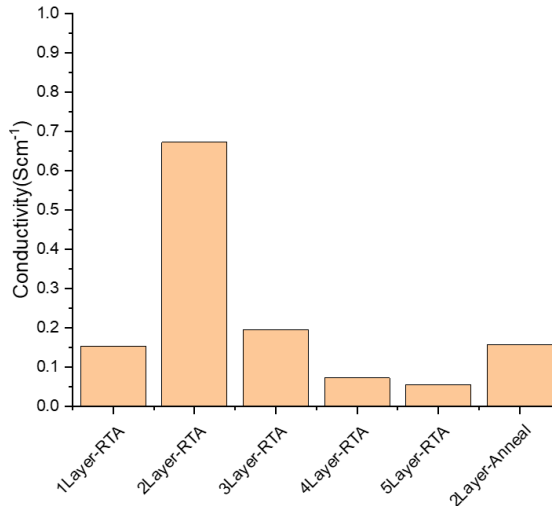


Figure 8. The conductivity of nc-AZO films with the various number of layers

Figure 8 illustrates the conductivity of nc-AZO with the various number layer with heat treatment by RTA at 930°C for 20second. It is found that 2 layers of nc-AZO films provide high conductivity approximately 0.7 Scm^{-1} . Meanwhile, The 2 layers of nc-AZO with traditional annealed at 500 Wang for 1hr. To provide low conductivity approximate 0.15 Scm^{-1} . 1 layer, 3 layer, 4 layer, and 5layer show low conductivity than other conditions.

Table 1 shows the comparison of photocurrent density with other research with various preparation methods. Table 1 details the comparison of photocurrent density values of other metal oxide films and spherical nc-AZO film. At low applied voltage (0.5 and 0.8V), It presents the high photocurrent density of spherical nc-AZO films are 830-fold. Thus, the formation of dense spherical nc-AZO at the film surface can enhance optoelectrical performance for perovskite solar cells and opto-sensor devices

The quality of spherical nc-AZO on quartz substrates are evidenced by the XRD patterns in Figure 9 and Figure 10. The crystal grain size of nc-AZO films was calculated by using Scherrer's relation as shown in the equation (3) (Zhu *et al.*, 2013)

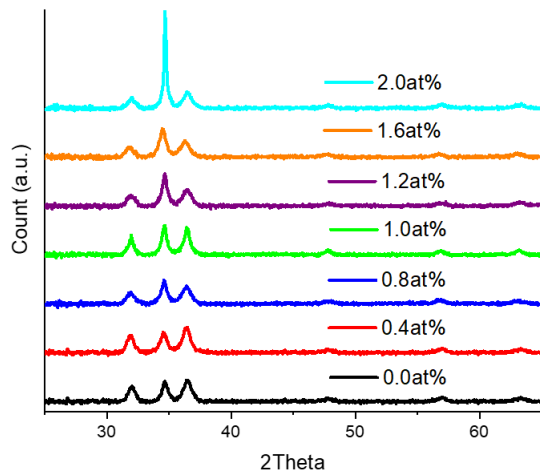
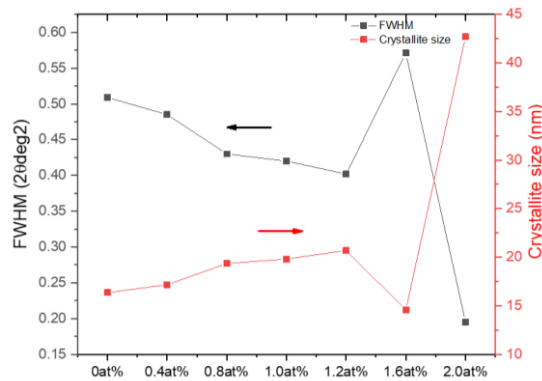
$$d = \frac{0.9\lambda}{B \cos \theta_B} \quad (3)$$

where d is the average crystal size
 λ is the wavelength of X-rays ($1.54059 \text{ \AA}$)
 B is the width at half of the peak height (FWHM)
 θ_B is the angle of Bragg

Figure 9 shows the XRD patterns of spherical nc-AZO films on quartz substrates with RTA-treated at the various Al dopants concentrations. This result showed that the structure of spherical nc-AZO films presents polycrystal with hexagonal wurtzite structure and all of the diffraction peaks match with that of ZnO (JCPDS #036-1451). The spherical nc-AZO films on quartz substrates with various Al dopants conditions obtained the ZnO peaks that identified to the plane $31.957^\circ(100)$, $34.635^\circ(002)$, $36.430^\circ(101)$, $47.645^\circ(102)$, $56.848^\circ(110)$, and $62.112^\circ(103)$ of diffractions and particular the dominant is the peak (002).

Table 1. Comparison of photocurrent of various oxide films

Material	J (mA/cm ²)	Preparation method	Reference
ZnO:Bi	97.02 (at 0.5V)	Sol-gel	Rattanawichai <i>et al.</i> , 2018
ZnO:Bi	632.95 (at 0.5V)	Sol-gel	Fangsuwannarak <i>et al.</i> , 2022
TiO ₂	0.55 (at 0.8V)	Plasma assisted chemical vapor deposition	Schoenhalz <i>et al.</i> , 2010
AZO/TiO ₂ nanocone array	0.9 (at 0.8V)	Atomic layer deposition	Randeniya <i>et al.</i> , 2007
ZnO nanorod	0.2 (at 0.8V)	hydrothermal	Hwang <i>et al.</i> , 2009
nc-AZO	829.19 (at 0.5V)	Sol-gel	This work

**Figure 9. XRD patterns of spherical nc-AZO films on quartz substrates****Figure 10. Relationship between FWHM and crystal size of spherical nc-AZO films doped with various Al concentrations**

The undoped ZnO film shows weak XRD peaks with the calculated crystal grain size of approximately 16nm. Meanwhile, the AZO films with increasing Al dopants concentration show strong XRD peaks at the (002) planes with

the absence of phase separation of Al₂O₃ peak (JCPDS CARD #10-173). The crystal grain size of nc-AZO films provided approximately 15 - 40 nm by calculation. When Al dopants concentration from 0.4at% to 1.2at%, FWHM of XRD peaks decreased, leading to an increase of grain size by calculation shown in Figure 10. These results have an agreement with effect on increasing crystal grain size as observed in the previous reports (Hu *et al.*, 2008; Nicolescu *et al.*, 2010; Yang *et al.*, 2015).

The lattice parameters *a* and *c* of nc-AZO films can be calculated by using the following equation (Krongarrom *et al.*, 2012)

$$d_{hkl} = \frac{1}{\sqrt{4(h^2+k^2+hk)/3a^2+l^2/c^2}} \quad (4)$$

Where *d_{hkl}* is the crystalline surface distance of *hkl* indices and *a* and *c* are the lattice parameters. In table 2, the undoped ZnO film lattice parameters are close to the parameters of (JCPDS 36-1451). The difference of ZnO films doped with Al concentrations gives higher *c* values than undoped ZnO films. ZnO films prepared on quartz substrates by sol-gel method provided higher *c* values (Ghosh *et al.*, 2004). The increasing parameter *c* value may be possible to substitute Al dopant into the Zn sites. The increasing Al dopants concentration until 1.2at% with the parameter *c* value slightly increased and with further increasing in doped level until 1.6at% the parameter *c* value decreased to lower. The parameter *c* value increased again in doped at 2.0at%. Therefore, 1.6at% Al concentration is the upper limit content for in-phase ZnO film in this study. The Relative intensity of (002) peak have slightly increased with increasing the Al-doped concentration.

Table 2. Lattice parameters and Relative intensity of (002) peak

Al dopant concentration (at. %)	Lattice parameters		Relative intensity of (002) peak
	a (Å)	c (Å)	
0.0	3.232	5.178	0.292
0.4	3.236	5.188	0.271
0.8	3.240	5.185	0.367
1.0	3.234	5.189	0.326
1.2	3.226	5.178	0.460
1.6	3.230	5.202	0.435
2.0	3.233	5.174	0.718

Conclusions

In this work, Spherical nanocrystalline AZO films successfully coated on quartz substrates have been prepared by the sol-gel method. The RTA-treatment process at a high temperature can improve good conductivity of nc-AZO films than using the annealing process. Surface morphology occurred such spherical nc-AZO particles size of 5-40 nm at 1.0at% and 1.2at% with 930°C for 20seconds. Moreover, wide energy band gap, E_g (3.27-3.30 eV) of nc-AZO film indicates high responsibility of wide range of photon energy which is a good property for the window layer of solar cells. RTA-treatment process, the spherical nc-AZO film provided uniform roughness, less reflectance, high photocurrent gain, and large grain size performances which can contribute to electron transport layer availability for perovskite solar cell, passivation layer for silicon solar cells, transparent conductor for batteries, and semiconductor devices such as pressure sensor potentiometric pH sensor and colorimetric sensor.

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