

THE STUDY OF ZINC OXIDE WITH SILICON DOTS THIN FILMS FABRICATED BY SPIN COATING TECHNIQUE FOR APPLYING AS EMITTER LAYER

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Abstract

A low-cost spin coating technique and rapid thermal annealing method have been utilized for the preparation of aluminum doped zinc oxide with Si dots thin film on the quartz substrates compared with zinc oxide doped bismuth thin film with low-temperature annealing. The dispersed Si dots particle in the film showed that these additional Si dots can be tunable material for light absorption applied in photovoltaic work. X-ray diffraction was used to study the nanocrystalline silicon and zinc oxide with the crystal grain size of ~100 and ~20 nm presented in the films, respectively. The optical result of the thin films shows that <20% of reflectance and >80% transmittance were presented in the film. The absorption edge of thin films that have occupied Si dots was expanded from the original zinc oxide which strongly absorbs UV wavelength (~380 nm) to near-infrared wavelength (~800 nm) depending on the amount of Si dots layer. For the electrical properties of the film, the two-probe method was used to observe the photocurrent gain under the illumination compared with the dark test and showed that the film can greatly respond to the light and provided a higher current density. Additionally, the increase in annealing temperature and amount of zinc oxide layer leads to the enhancement of the current density. Hence, these zinc oxide thin films can be an alternative candidate for the tunable absorption emitter layer of solar cells and can be applied for other optoelectronic devices.

Keywords: Zinc oxide doped aluminum; Zinc oxide doped bismuth; Silicon powder; Thin film

Introduction

Solar cells are semiconductor devices that can directly convert sunlight to electric power, most of them are fabricated from silicon (Si) material which has an energy band gap of 1.1 eV and the Si cell can provide maximum efficiency of 30% due to the

detailed balance limit of efficiency (Shockley and Queisser, 1961). The concept of tandem cells can apply to be on top of the solar cell to improve light absorption and efficiency because the different band gaps that are higher than Si can convert

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a narrow range of light energies to their energy gap (Green, 2002)

Zinc oxide (ZnO) is well known for applying much attention to research due to the direct band gap of n-type material and its optical properties, it has a broad energy band of 3.37 eV, which can especially respond to the ultraviolet (UV) wavelength (Kołodziejczak-Radzimska and Jesionowski, 2014). The transparency of ZnO film can provide a higher wavelength of light passing through the film easily. The ZnO thin film process has been carried out by many methods such as sputtering (Ramadan *et al.*, 2022), plasma-enhanced chemical vapor deposition (Barankin *et al.*, 2007), spray pyrolysis (Grothe *et al.*, 2020), and the sol-gel method Rattanawichai *et al.*, 2018). In addition, the ZnO can be a composite material with other materials to improve their electrical properties in supercapacitors application (Wang *et al.*, 2018), to enhance the thermal conductivity and dielectric constant (Fang *et al.*, 2015), and to be the ferroelectric ZnO nanowire polymer composite layer for instantaneous dynamic motion sensor (Shin *et al.*, 2017). Moreover, from our previous work with nanocrystalline ZnO doped with Bi films and Si powder in the matrix of ZnO doped Bi has also been reported about their properties and emitter layer on solar cell (Krongarrom *et al.*, 2012; Fangsuwannarak *et al.*, 2013; Rattanachan *et al.*, 2013; Fangsuwannarak *et al.*, 2021).

In this work, we have improved and studied the silicon dots in matrices film of zinc oxide doped bismuth and aluminum and applied Si powder from waste silicon wafers in the solution of zinc oxide sol-gel as the source of Si dots thin films. The comparison between normal annealing of 550°C for 2 h. and rapid thermal annealing of 930°C for 30 seconds has been reported. The Si dots thin films convinced the improvement in the electrical properties and absorbance mechanism

Materials and Methods

Si powder as a material for Si dot films was obtained from waste Si wafer transmogrified by grinding technique. The average small particles of Si powder of 4 µm were accomplished by conventional ball mill planetary QM2L which have detailed in our previous work (Fangsuwannarak *et al.*, 2021; Laohawiroj *et al.*, 2018). The substances for the synthesis of the aluminum doped zinc oxide and bismuth-doped zinc oxide consist of zinc acetate dehydrate (ZnAc, Zn-(CH₃COO)₂·2H₂O, 99.5%) as a ZnO source, ethylene glycol (EG, CH₂OHCH₂OH, 99.5%), mono-ethanolamine (MEA, NH₂C₂H₄OH, 99%),

Bismuth nitrate pentahydrate (Bi, Bi(NO₃)₃·5H₂O, 98%) as a bismuth dopant source, aluminum chloride hexahydrate (AlCl₃, AlCl₃·6H₂O) as an aluminum dopant source, ethanol (EtOH, CH₃CH₂OH), isopropanol (IPA, (CH₃)₂CHOH) and hexadecyl-trimethyl-ammonium bromide (CTAB, C₁₉H₄₂BrN, 96%) as a surfactant.

The transparent ZnO solutions were separated into 2 solutions depending on the bismuth and aluminum dopant sources. Zinc oxide doped bismuth (ZnO:Bi) solution preparation was described in our previous work (Fangsuwannarak *et al.*, 2021). ZnAc of 0.7 M was dissolved in a solution of EG-MEA and molar ratio of Zn:MEA was kept at 1:1. The mixture was stirred at 80°C for 10 min and then added bismuth nitrate pentahydrate of the concentration 0.2 atom %. The mixture was continuously stirred at 80°C for 1 h and then slowly dropped 0.823 mM CTAB for dispersing quality enhancement of the obtained ZnO:Bi films. The solution was continuously stirred for 30 min to ensure homogeneity of the complete sol-gel then soaking solution at room temperature for 24 h. Zinc oxide doped aluminum (ZnO:Al) solution was described in Salam *et al.* (2013) work. Firstly, ZnAc of 0.2 M was dissolved in a solution of IPA-MEA and kept the ratio of MEA:Zn equal to 0.75:1, then, the solution was stirred at 60°C for 30 min. After that, the 0.2M of ALCL dissolved in EtOH was added to the ZnO-IPA-MEA solution to receive 1% atom of Al. Then, the solution was continued stirring at 60°C for 30 min and the clear solution was aged at room temperature for 24 h. For Silicon dots solution, ZnO:Bi solution and ZnO:Al solution were a matrix for Si powder. The amount of the Si:ZnO solution was kept at 1 g : 66.67 ml and well dispersed in an ultrasonic bath for 10 min. After that, spin coating technique was used for depositing the thin film with 2-step coating by 100 rpm for 15 second and 2500 rpm for 30 second, respectively. Thin films were dried at 200°C for 30 min and then some thin films were repeated deposition and dried to obtain 3 layers of ZnO thin film. The difference annealing of the film were varied, for instance, annealing at 550°C for with 1 hr for ZnO:Al and 2 hr for ZnO:Bi and rapid thermal annealing at 930°C for 30 second.

The prepared ZnO and ZnO with Si powder films were measured the thickness by optical profiler (Bruker) and characterized the surface morphology, crystalline structure and crystal grain size of ZnO and Si, optical properties and electrical properties by field emission scanning electron microscopy ((FESEM), Auriga Carl Zeiss), X-ray diffractometer (Bruker D8 corporation), UV-VIS-NIR spectrophotometer (LAMBDA 950 of PerkinElmer Inc.,) and 2400 electrometer (Keithley), respectively

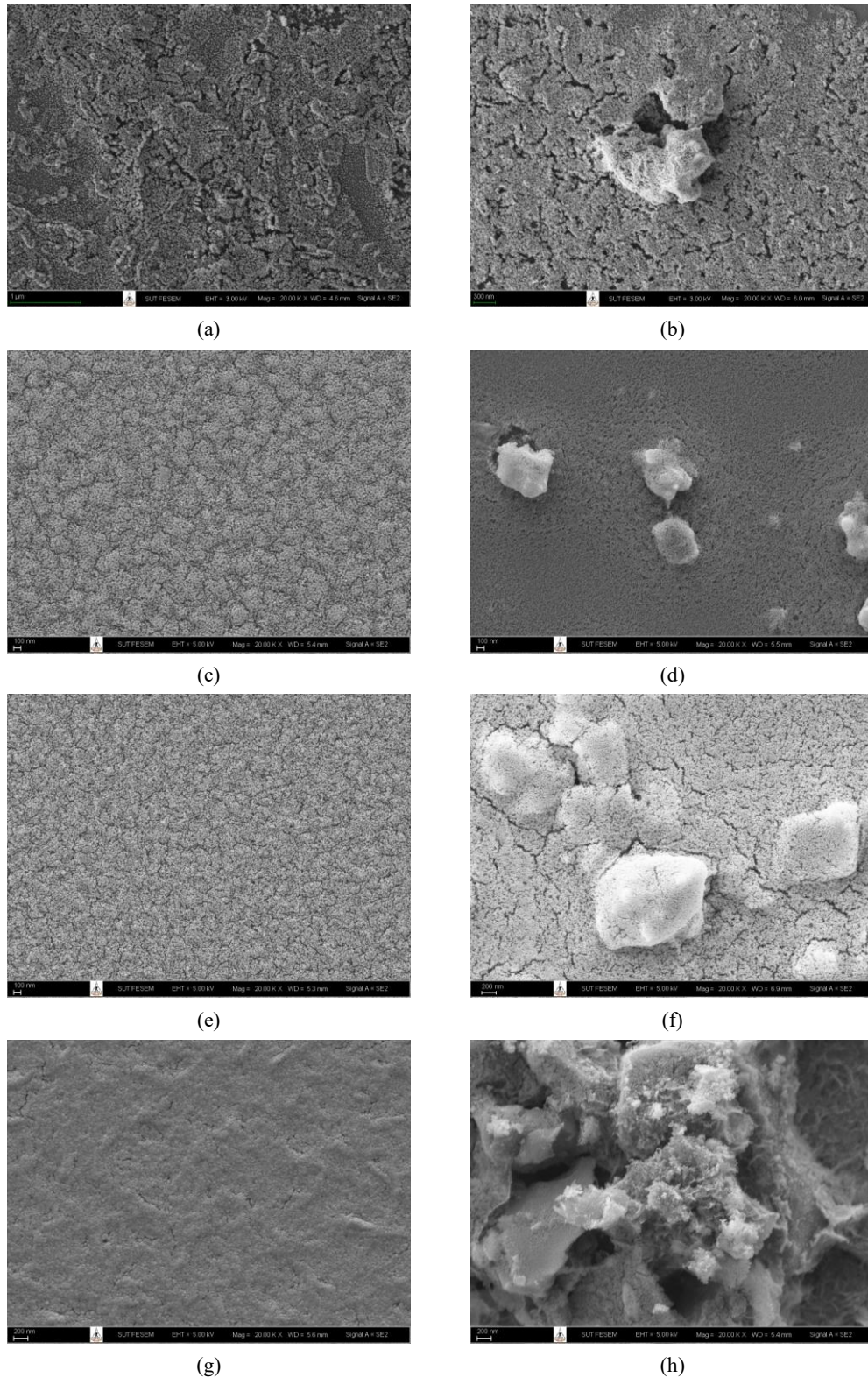


Figure 1. FESEM images with of ZnO and Si dots thin films of (a) ZnO:Bi 1L 550, (b) ZnO:Bi:Si 1L 550, (c) ZnO:Al 1L 550, (d) ZnO:Al:Si 1L 550, (e) ZnO:Al 1L 930, (f) ZnO:Al:Si 1L 930, (g) ZnO:Al 3L 930 and (h) ZnO:Al:Si 3L 930

Results

Figure 1 showed the 20k magnification of surface morphology of the ZnO doped bismuth and aluminum with and without Si powder in different annealing temperature of 550°C and 930°C and in different layer of 1 and 3 layers on quartz substrate. In Figure 1(a) and Figure 1(c), FESEM image show the surface morphology of the ZnO thin film prepared by different dopant source as bismuth and aluminum with annealing temperature of 550°C, respectively, it showed that the ZnO particles of ZnO:Al cover the entire area on quartz substrate meanwhile the ZnO particle of ZnO:Bi presents some void area because the substance of EG and IPA have different viscosity. The different temperature annealing of 550°C and 930°C for 1 layer as shown in Figure 1 (c) and (e) expressed the uniform nano spherical ZnO:Al particles, while for 3 layers of ZnO:Al in Figure (g) shown the homogeneous agglomerate of spherical ZnO:Al. For Si dots films in both ZnO:Bi and ZnO:Al as shown in Figure 1(b), (d) and (f) appear the Si particles covered by nano spherical ZnO and scattered on the film with Si size is less than 2 μm , however for 3 layers of Si dots in Figure 1 (h) show the agglomerate Si alluvion. The average thickness of the thin films in the Table 1 exhibit that the ZnO doped bismuth and aluminum film for 1 and 3 layers have the average thickness about 65 and 120 nm, respectively, while the Si dots films of 1 and 3 layer have the average thickness about 120 and 170 nm, respectively

Figure 2(a) shows the XRD pattern of prepared ZnO and Si dot thin films. XRD pattern of Si was expressed in the sample that add Si powder, corresponding to a reference pattern of Si crystalline structure (JCPDS 00-027-1402) which compose of 2θ around 28.4°, 47.3° and 56.1° with orientations along (111), (220) and (311) planes, respectively. It is noted that the intensity of crystalline Si peak increase due to the amount of possessed Si in the film. Figure 2(b) shows the magnifying XRD pattern of 2 theta in range between 30° - 40° in order to interpret the crystal structure of different condition of ZnO thin films. ZnO peaks were observed at position about 31.8°, 34.5° and 36.5° with orientations along (100), (002) and (101) planes, respectively, which corresponding to a reference pattern of spherical ZnO crystal (JCPDS 36-1451). For the ZnO samples were annealed with low temperature of 550°C, it shows that ZnO:Bi thin films obviously expresses the 3 peaks of spherical ZnO crystal while the ZnO:Al thin film appears small pattern, this indicate that the ZnO doped aluminum hardly form into the nano crystal for low temperature annealing. However, the ZnO:Al thin film for 1 and 3 layers that was annealed at high temperature of 930°C occurs the ZnO crystal pattern higher than annealing temperature of 550°C, this shows that the annealing temperature is significant for forming the nano crystal of ZnO.

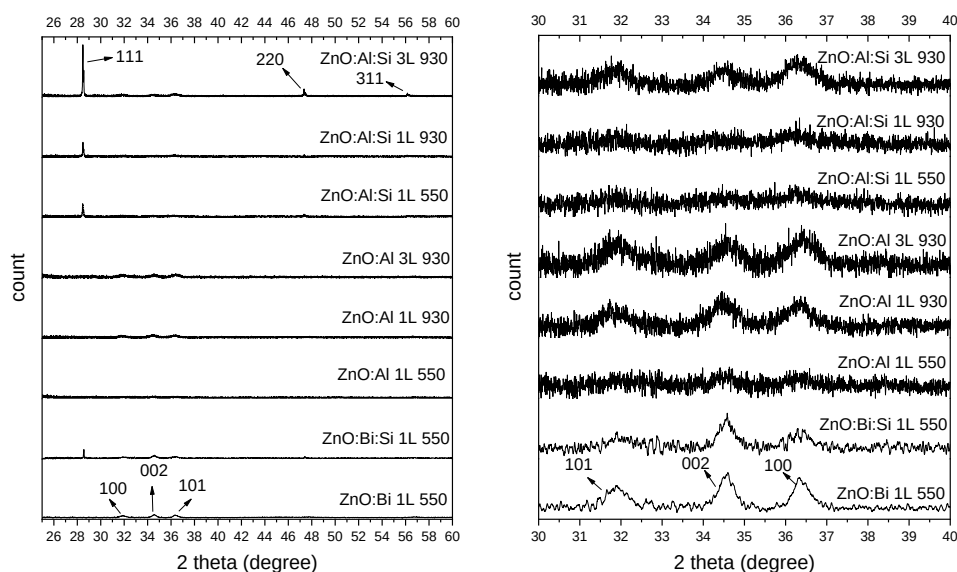


Figure 2. XRD pattern of ZnO thin films in range of 2 theta of (a) 25° - 60° and (b) 30° - 40°

The simple cubic structure of Si and hexagonal wurtzite structure of ZnO of (111) and (002) crystallographic planes, respectively, can be verified the average crystal grain size by Scherrer's relation equation in equation (1) and the calculated results are presented in the Table 1. It explains that the average crystal grain size of Si is between 90 - 107 nm, it is note that the crystal grain size of 3 layers of ZnO:Al:Si thin film does not increase with the number of the layer. The temperature annealing of 550°C of ZnO sample shows the crystal grain size of ZnO is smaller than temperature annealing of 930°C of ZnO sample. However, ZnO crystal grain size of Si dots film of 1 layer with annealing temperature of 550°C and 930°C is resembling value. Moreover, the crystal grain size of 3 layer of Si dots film is bigger than the 1 layer.

$$d = \frac{0.9\lambda}{B \cos(\theta_B)} \quad (1)$$

Where d is the average crystal grain size (nm), λ is the X-ray radiation wavelength with 0.154 nm, B is the full width half maximum (rad) and θ_B is the Bragg diffraction angle of XRD peak (degree)

The small shift of (111) peak and (002) broad peak refers to the change of lattice constants for simple cubic structure of Si and hexagonal wurtzite structure of ZnO, respectively. The strain along c-axis (ε) was determined to evaluate the change of lattice constants which given in equation (2) and the results are reported in Table 1. The result of strain along c-axis express the compressive strain (negative sign) that occurs for every sample both ZnO thin film and Si dots thin films, it is due to the interstitial impurity doping atom and annealing temperature (Rattanachan *et al.*, 2013).

$$\varepsilon = \frac{C - C_0}{C_0} \quad (2)$$

Where C is the lattice parameter of the film calculated from XRD data, C_0 is the lattice parameter of the pure Si crystal ($C_0 = 5.43 \text{ \AA}$)

(Hubbard *et al.*, 1975) and ZnO crystal ($C_0 = 5.207 \text{ \AA}$) (Morkoç and Ü. Özgür, 2009)

Figure 3(a) and (b) express the light reflection and light transmission spectra in wavelength of 200-1500 nm for different condition of ZnO thin film with and without Si powder, respectively. It is shown that the reflectance values of all sample are less than 15% in all region, except the ZnO:Al:Si with 3 layers that has the value of reflectance more than 15% in infrared region. The transmittance of the ZnO thin film with and without Si powder of 1 layer express the light can transmit through more than 80% in visible and infrared region. Note that the increase of Si powder in the film can conceal the film and reduce the transmittance in all wavelengths.

$$\alpha = \frac{1}{t} \ln \left(\frac{\sqrt{(1-R)^4 + 4T^2 R^2} - (1-R)^2}{2RT} \right) \quad (3)$$

Where t is the average thickness of the film, R and T are the reflectance and transmittances values, respectively.

The optical absorption coefficient can be calculated by relationship from equation (3) and can be plotted the curve as shown in Figure 3(c). Note that the Silicon dot film is composite material of Si powder in ZnO matrix, hence the optical bandgap can be implied as the estimation of the absorption edge (Pankove, 1976). The ZnO:Bi and ZnO:Al thin films with different temperature annealing have an absorption edge around 3.3 eV, whereas the 3 layers of ZnO:Al can be slightly shifted to 3.2 eV. This indicate that group of these ZnO thin film efficiently absorb the UV light. In addition, the Si dots films of ZnO:Al:Si samples was observed that the absorption edge can be expanded to visible region and near infrared region depending on the number of Si dots layers. Nevertheless, the absorption edge of ZnO:Bi:Si sample is still at UV region. Moreover, the refractive index curve as shown in Figure 3(d) can compute from the equation 4 and the result at the wavelength 600 nm is reported in Table 2. The result suggests that an increase of

Table 1. Crystalline size and strain along c-axis of Si and ZnO

Condition	Thickness (nm)	Crystal grain size (nm)		Strain along c-axis	
		Si (111)	ZnO (002)	Si (111)	ZnO (002)
ZnO:Al 1L 550	68.43 ± 15.81	-	11.09	-	-0.327
ZnO:Al 1L 930	60.57 ± 0.96	-	19.36	-	-0.014
ZnO:Al 3L 930	120.46 ± 8.13	-	20.36	-	-0.372
ZnO:Al:Si 1L 550	124.36 ± 9.65	90	11.84	-0.024	-0.054
ZnO:Al:Si 1L 930	130.29 ± 20.03	106	10.38	-0.103	-0.092
ZnO:Al:Si 3L 930	176.13 ± 10.59	107	15.89	-0.096	-0.392
ZnO:Bi 1L 550	67.30 ± 15.65	-	18.90	-	-0.291
ZnO:Bi:Si 1L 550	105.42 ± 19.25	103	25.10	-0.336	-0.481

number of layers of the film results in an increase of refractive index and the refractive values are less than 3, hence this film can be applied to be as an anti-reflection coating (ARC) layer and emitter layer for solar cell application.

$$R = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2} \quad (4)$$

Where n is the refractive index of the film, k is the extinction coefficient that can define as $k = \alpha/4\pi$

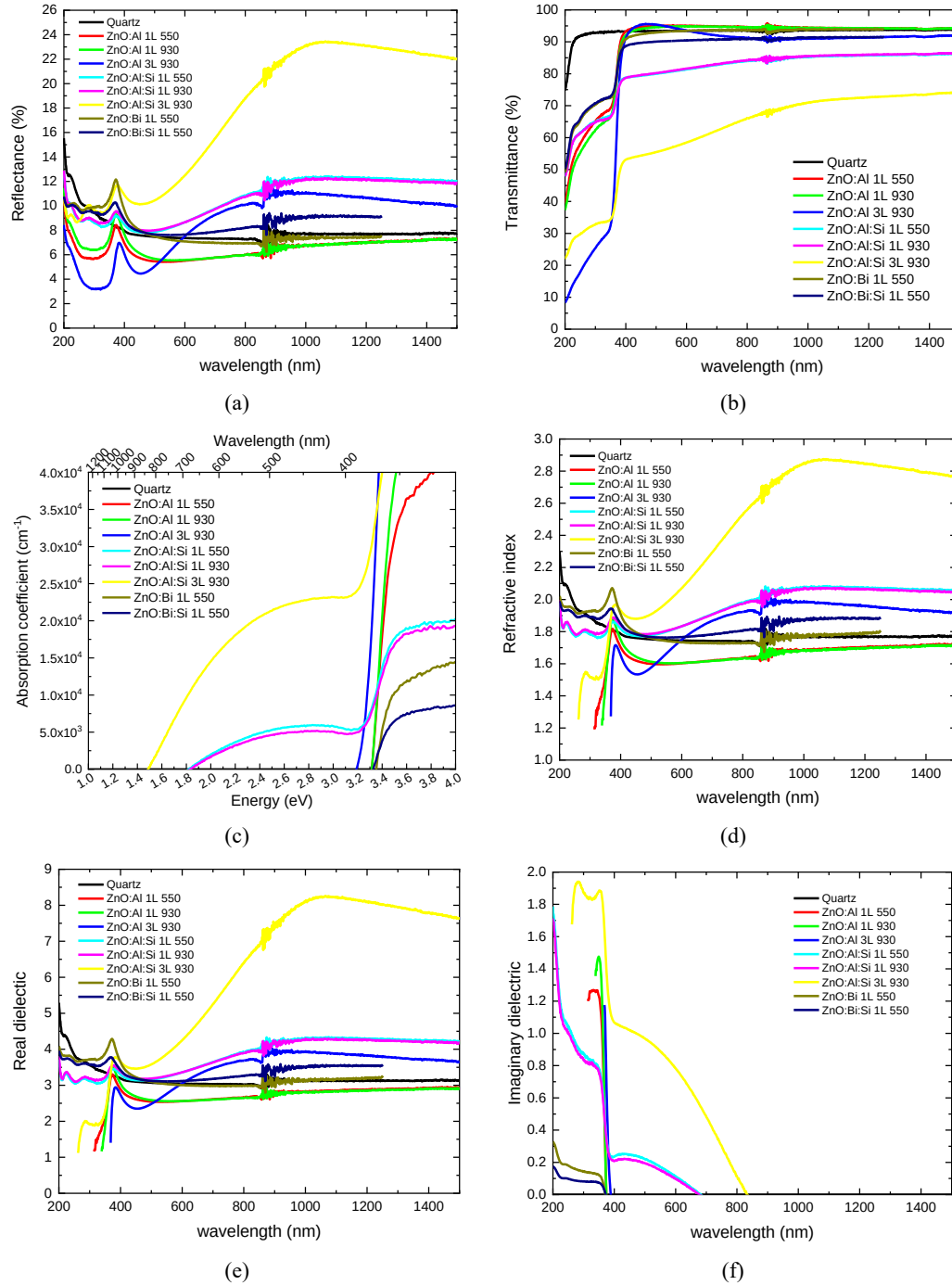


Figure 3. (a) Reflectance, (b) transmittance, (c) absorption coefficient, (d) refractive index, (e) real dielectric and (f) imaginary dielectric of ZnO thin films and Si dots thin film

Dielectric constant can be separated into 2 parts to real dielectric (ϵ_r) and imaginary dielectric (ϵ_i) as a function of wavelength which are performed in Figure 3(e) and Figure 3(f) and can calculate from equation (5) and (6), respectively.

$$\epsilon_r = n^2 - k^2 \quad (5)$$

$$\epsilon_i = 2nk \quad (6)$$

The value of real dielectric expresses the dispersion of the electromagnetic waves travel inside the materials while the imaginary dielectric related to the energy absorption from electric field from dipole motion (Hassanien, 2016). The real dielectric value of the thin films is more than the imaginary dielectric, this indicates that the light can easily disperse in the film. However, at UV wavelength of 200-380 nm, the ZnO:Al films reports the high value of the imaginary dielectric, it means that the UV light are dramatically absorbed by the free carrier in the films.

Figure 4(a) shows the JV-curve from 2-probe method that the films were applied voltage from -10 to 10 V under the dark test and illumination test condition and measured the current. The linear curve expresses the ohmic behavior between the heterojunction of the metal and thin film. The different carrier generation under the illumination test was exhibited in Figure 4(b), the current density of ZnO thin films increases when they were exposed under the light, in addition this amount of Si powder in the Si dot film did not lead to the light induced degradation due to the increment of trap state between the Si powder and ZnO matrix (Wolkin *et al.*, 1999), hence they can generate the carrier and reduce the resistivity in the film under the illumination condition as shown in Figure 4(c) with the result of the photocurrent gain value from ratio of current density between illumination and dark test. The relationship of the resistivity (ρ) was calculated in equation (7). The 3 layers of ZnO:Al provides the small values of resistivity if compare with 1 layer of ZnO:Al and ZnO:Bi because the electron mobility was improved from the increase of ZnO crystal grain size that can reduce the grain boundaries in the film and also the improvement of the film surface morphology (Khan *et al.*, 2017).

$$\rho = \frac{V}{J \times l} \quad (7)$$

Where V is the applied voltage (V), J is the current density (A/cm^2) and l is the distance between the metal contact that applied voltage (cm)

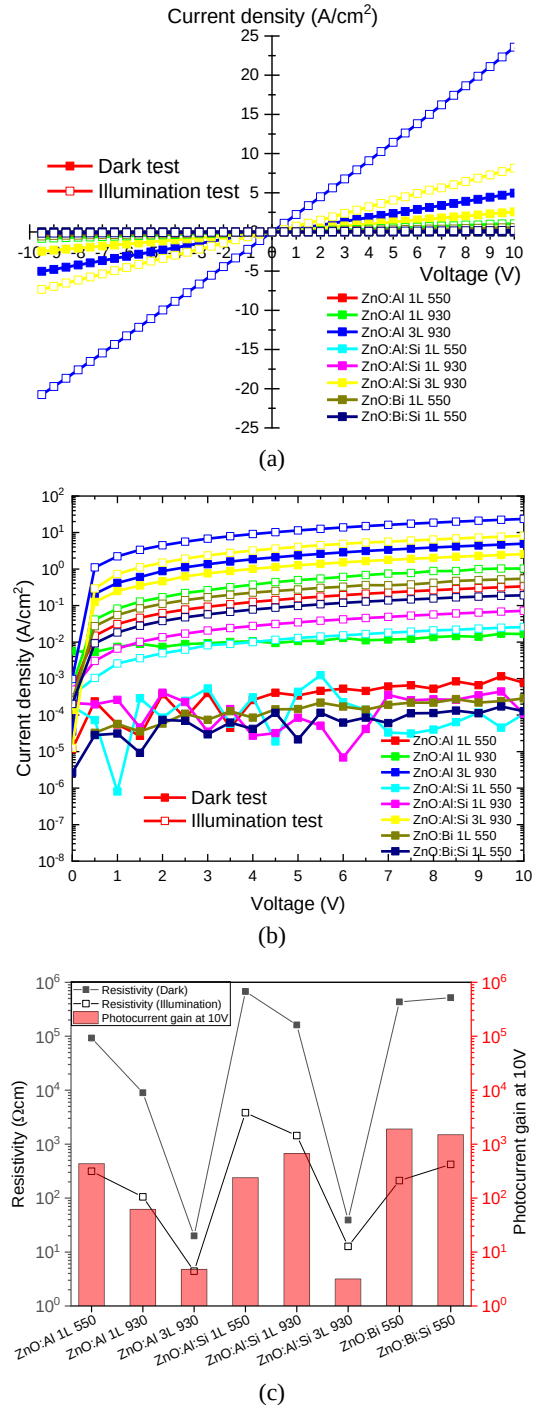


Figure 4. JV curve of thin films in (a) linear and (b) semi-log scale. and (c) the curve of resistivity and photocurrent gain at 10 V of thin films

Table 2. Absorbance edge and refractive index at wavelength 600 nm of Si and ZnO

Name	Absorbance edge		refractive index
	nm	eV	
ZnO:Al 1L 550	374	3.315	1.609
ZnO:Al 1L 930	374	3.315	1.615
ZnO:Al 3L 930	389	3.190	1.747
ZnO:Al:Si 1L 550	685	1.810	1.833
ZnO:Al:Si 1L 930	678	1.830	1.831
ZnO:Al:Si 3L 930	832	1.490	2.060
ZnO:Bi 1L 550	370	3.350	1.733
ZnO:Bi:Si 1L 550	373	3.325	1.765

Conclusions

Si dots films have been successfully fabricated from Si ink which synthesized from transmogrified waste Si wafer into Si powder with ZnO doped Al and ZnO doped Bi solution. The simple method as spin coating technique has been used for thin film depositing on quartz substrate and the thin films were annealed with different processes of normal annealing and rapid thermal annealing. The surface morphology of the film with ZnO doped with Al express the dispersion of ZnO particle smoother than ZnO doped Bi because of its viscosity in the solution. The improvement of grain boundary of ZnO particle can be done with RTA method and depositing the film more than 1 layer. The crystal structure of both ZnO:Bi and ZnO:Al thin films express the same peak with XRD pattern with the crystal size between 10 - 25 nm depending on the temperature annealing. Although the films were annealed with different method and doped with different element, they still attribute the compression strain. The ZnO films have their properties to absorb the UV wavelength but the existence of Si powder in Si dots film can improve the energy absorption in visible wavelength that reported from absorption coefficient. The result from illumination test with two probe method provides the photocurrent gain which mean that the ZnO and Si dots films are less defect states and disappear the light induced degradation. Hence, the ZnO thin films with Si powder or Si dots films can be applied on the emitter layer that need to improve the absorption properties of the solar cell.

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