Fabrication of UV photoconductive sensor using Aluminium doped ZnO nanorod arrays anneal in different environment

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***Abstract*-** Aluminium doped Zinc Oxide (ZnO) nanorod arrays anneal in different environment and different oxygen flow rate were fabricated of Al doped ZnO nanorod arrays prepared using sonicated sol-gel immersion. The FESEM are used to measure the substrate of the optical properties which surface morphology and thickness which has been seen that the thickness nearly to 600 nm and the diameter on the top surface of the nanorod is around 40 to 60 nm. The optical transmittance and absorbance was measure using UV-vis-NIR. Nanohole occur during the air annealed much more than oxygen annealed and vacuum annealed. The characteristic of the Al doped ZnO nanorod arrays seem give good performance according to the sensitivity under UV photoconductive sensor. These experiment are study that the oxygen occur through the increasing the flow rate compare to the other environment such as air and vacuum which annealed in oxygen give more sensitivity of the fabricated substrate due to expose of light from the UV photoconductive sensor.

I. INTRODUCTION

Nowadays, metal oxide semiconductor already used in many application including solar cells [1], gas sensor [2], light-emitting diodes [3]. Zinc oxide (ZnO) is one of the metal oxide semiconductor area studies due to the chemical and physical properties which ZnO have wide band-gap energy of 3.3 eV and a large exciton binding energy of 60 meV at room temperature. Beside, ZnO also has many advantages such as cheaper, easy to fabricated, alternatively potential of emerging other than GaN in optoelectronic applications [4].

ZnO nanorod arrays based ultra-violet (UV) photoconductive sensor is seen interesting due to physical and chemical properties that had been told early and nanorod arrays itself have a potential photodetectors infrastructure because nanoscale feature of arrays and nanorod surface area that asymmetric response to an incident light of UV photoconductive sensor that could be improve the sensor performance. There are some of techniques can be used to prepared ZnO nanorod arrays such as chemical vapour deposition (CVD) [5], metalorganic chemical vapour deposition (MOCVD) [6], pulsed laser deposition (PLD) [7] and sol-gel [8]. Sol-gel has define as thepreparation process material due to condensation and polymerization which this technique has some advantage rather than the other method which is low cost, dispersed to a solvent to form low viscosity of the solution in short period time, easy to quantify the incorporation of some trace elements evenly to achieve uniform doping on a molecular level, and chemical reaction will be easily occur only at a low reaction of temperature of annealing process. Generally annealing process is one of the important parts of semiconductor growth. In this paper study we prepared an Al doped ZnO nanorod arrays using sonicated sol-gel immersion method on the glass substrate that annealed in different environment and different flow rates of oxygen for UV photoconductive sensor application and its effect on ZnO properties have been investigated.

II. METHODOLOGY

A. Cleaning the Substrate

The substrate must be cleaned first because it should be considered that all the substrate already contaminated with dust and fingerprint. Some of step can be apply to make sure the substrate totally clean before it dry, the chemical such as acetone, methanol/ethanol and de-ionized (DI) water are used for this cleaning process. The time is set 20 minute and temperature is 50°C, all substrate must be sonicated with an ultrasonic water bath for each of chemical solution. Nitrogen gas was used to remove the small contamination particle at the surface of substrate after cleaning process.

B. Thin Film Deposition

Sol-gel spin coating method are used to prepared the seed catalyst for the substrate. A 100ml solution was prepared using zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) as a precursor, aluminum nitrate nonahydrate ($Al(NO_3)_3.9H_2O$) as a dopant source, monoethanolamine (MEA, $C_2H_7N_{14}$) as a stabilizer and

2-methoxyethanol as a solvent and stirred at 80°C of temperature and duration in 3 hour. After that the
solution was stirred and aged for 24 hours before deposition process. The Al doped ZnO thin film using spin-coating was performed for deposition process at room temperature using 10 drops of solution per deposition with a speed of 3000 rpm for 60 s. Each time after deposition process, the films were heated in air using a furnace at 150°C for 10 minute to evaporate the solvent. After repeating 5 times of the coating, the films were annealed at 500°C for 1 hour in air using a furnace.

C. Sonicated Sol-Gel Immersion Method

Al doped ZnO nanorod arrays was prepared using sonicated sol-gel immersion method on the glass substrate coated with a ZnO thin film using a solution 0.1M zinc nitrate hexahydrate [$Zn(NO_3)_2.6H_2O$], 0.001M aluminium nitrate nonahydrate [$Al(NO_3)_3.9H_2O$], and 0.1M hexamethylenetetramine (HMT; $C_6H_{12}N_4$). This solution was sonicated at 50°C of temperature for 30 minute using ultrasonic water bath. This solution was then stirred and aged for 3 hour at room temperature. After that the thin-film-coated glass substrate was placed horizontal in the Schott bottle and the solution was poured into each of the Schott bottle. The sealed Schott bottle was then immersed in a 95°C hot water bath for 50 minute. After the immersion process, the glass substrate were cleaned with de-ionized (DI) water before heated at 150°C using furnace for 10 minute.

D. Annealing Process

The annealing process was prepared using HV.GSLI 1700 high temperature furnace. Oxygen annealing are conduct with put the substrate into the furnace than set the temperature at 500°C, pressure gas of oxygen is 0.25Pa (120 Bar), and the flow meter is 0.2 L/min (200sccm). The oxygen annealed take several hour of that process and after done the process it should be follow by cooling process around 1 hour. Repeat the same step for the other substrate with only change the flow meter to 0.4 L/min, 0.6 L/min, 0.8 L/min and 1.0 L/min. Next for air annealing was take part with annealed the substrate in the chamber furnace that contain air ambient and set the temperature of 500°C at 1 hour. Meanwhile for the vacuum annealed with annealed the substrate in vacuum ambient that set at 3 torr and set the temperature at 500°C and several hour a take to annealed the substrate.

E. Deposition Metal Contact

The aluminum contact was deposited on to the nanorod array film using electron thermal evaporation with a thickness of aluminum is 60 nm to produced a metal-semiconductor-metal (MSM) type of UV photoconductive sensor which the separation metal contact was similar to all substrate by 2 mm.

F. Chacterization

The characterized of the substrate of Al doped ZnO nanorod arrays are analyzed by field-emission scanning electron microscopy (FESEM; Zeiss Supra 40VP) to measure the surface morphology and thickness of the Al doped ZnO nanorod. Beside the optical transmittance and absorbance properties was determine using UV-visible-near infrared (UV-vis-NIR) spectrophotometry (Varian Cary 5000). Meanwhile for the UV photoresponse measurements of the fabricated sensor was measure using UV photocurrent measurement (Keithley 2400) which operated at 365 nm with a bias voltage of 10 V and a power density of 750 uW/cm^2.

G. Flowchart of Methodology

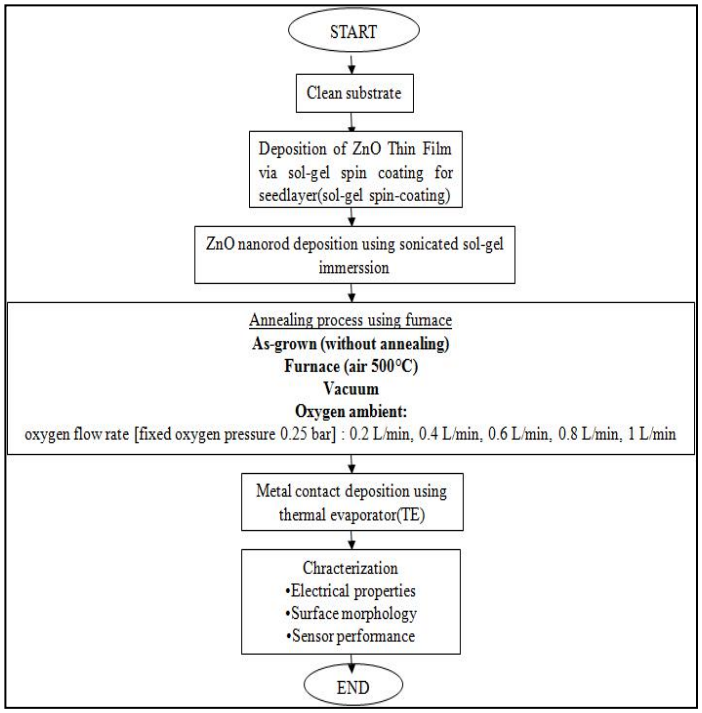

Figure 1: Flowchart of the methodology.

III. RESULTS AND DISCUSSION

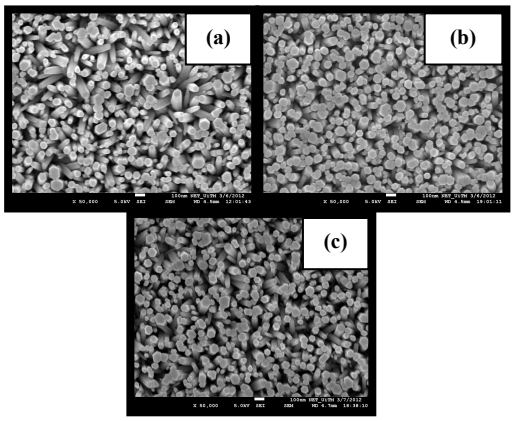

Figure 2(i): FESEM images of Al doped ZnO nanorod arrays annealed in different environment:
(a) oxygen [1 L/min], (b) air, and (c) vacuum.

Figure 2(i) show the result of surface morphology of Al doped ZnO nanorod annealed in different environment for oxygen [1 L/min], air, and vacuum. The images of field emission scanning electron microscop (FESEM) are set for the magnification of 50 k through the entire sample with the voltage 5.0 kV. The image show at figure 2(i)(a) is annealed in oxygen flow rate at a 1 L/min has been seen that the nanorod structure due to crystallinity and uniformity is more better either than annealed in air and vacuum. The average diameter size that annealed in oxygen is 51.62 nm. Meanwhile average diameter size annealed in air at 500°C is 60.93 nm and for the vacuum, the average diameter size is about 57.7 nm. If close look deeper using FESEM at these three annealed environment, it was observed that nanohole are exist in high quantities for air-annealed rather than oxygen-annealed and vacuum-annealed. The nanohole exist due to annealed at high temperature and these nanohole surface itself are not stable due to the crystal defect and dangling bond such as zinc interstitial and oxygen vacancies. Beside, according to the density of nanorod arrays annealed in oxygen show more high and more vertically growth with closed contact to each other than annealed in air and vacuum.

Figure 2(ii) show the result of surface morphology of Al doped ZnO nanorod arrays for seed-layer, as grown and oxygen annealing with different flow rate from 0.2 L/min to 1.0 L/min. The FESEM magnification and voltage is set still same with the previous, at the seed layer image show at figure 2(ii)(a) that the substrate not perfectly form well of nanorod arrays because at this level the substrate not sonicated sol-gel immersion yet with the average diameter size is about 15.65 nm. After the sol-gel immersion process take part the changing of the structure show at the as-grown at figure 2(ii)(b), the hexagonal shape on the top surface of the substrate begins to form and the structure become nearly become nanorod array that is a good crystallinity with the average diameter size is about 49.62 nm.

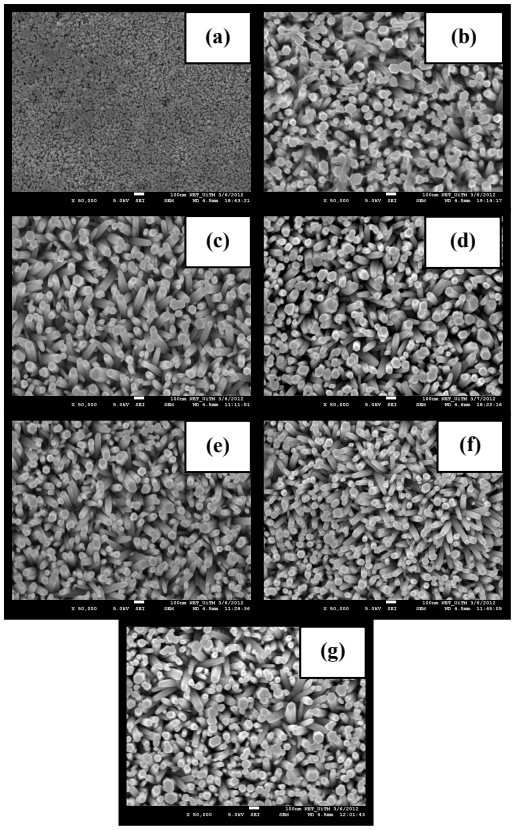

Figure 2(ii): FESEM images of Al doped ZnO nanorod arrays of (a) seed-layer, (b) as-grown, and oxygen annealed with different flow rate: (c) 0.2 L/min, (d) 0.4L/min, (e) 0.6 L/min, (f) 0.8 L/min, and (g) 1.0 L/min.

Meanwhile for the figure 2(ii)(c, d, e, f, g) show that the substrate anneal in oxygen with different flow rate at 0.2 L/min, 0.4 L/min, 0.6 L/min, 0.8 L/min, and 1.0 L/min with the

average diameter size is 49.77, 57.66, 43.42, 41.6, 51.62 nm. At

for each of the substrate that anneal in oxygen. It can observed that the increasing of the oxygen flow rate produce more density of nanorod arrays that can seen at of 1.0 L/min is high density rather than 0.2 L/min. Beside the hexagonal shape of nanorod structure become more sharp of edge and high uniformity. At the 1 L/min flow rate oxygen also show that the nanorod array growth more vertically and most closed contact of each other. The estimated range of nanorod diameter size annealed in oxygen with different flow rate is in between 40 to 60 nm and the determining diameter size of nanorod is due to grain size of seed-layer [9]. From the FESEM image, it could be suggest that the annealing process in oxygen with different flow rate has increasing in crystallinity and uniformity which at 1 L/min of oxygen flow rate is the optimum producing of nanorod arrays.

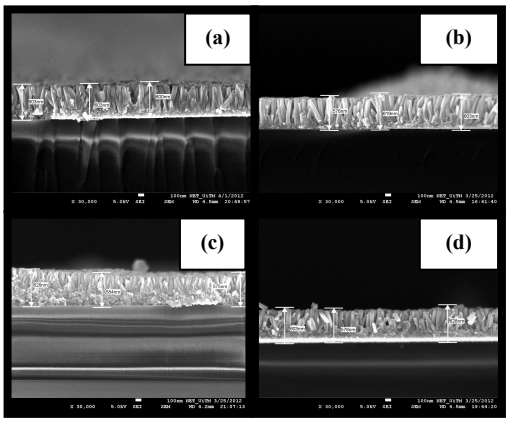

Figure 3(i): Cross-sectional images of Al doped ZnO nanorod arrays of (a) as-grown and oxygen annealed with different flow rate: (b) 0.2 L/min, (c) 0.6 L/min, and (d) 1.0 L/min.

Figure 3(i) show the cross-sectional images of Al doped ZnO nanorod arrays for as-grown and oxygen annealing with different flow rate that choose randomly at a rate of 0.2 L/min, 0.6 L/min and 1.0 L/min to measure the nanorod thickness for each of that substrate using FESEM. The cross-sectional of as-grown has an average nanorod thickness is 602 nm. Beside, for the average nanorod thickness of oxygen annealed with different flow rate such as 0.2 L/min, 0.6 L/min, and 1.0 L/min is 602, 596, and 603 nm. While for the figure 3(ii) is result for the cross- sectional images of Al doped ZnO nanorod arrays annealed in different environment such as oxygen [1 L/min], air, and vacuum. It observed that the result of this cross-section show that for the side view of nanorod structure for vaccum produce much more porosity in between this level it has been seen that the structure of nanorod arrays become more smooth and better crystallinity
each nanorod structure compare to the other which the nanorod structure that annealed in oxygen is vertically closed to each other and for annealed air show the nanorod structure also growth well but not in same shape of nanorod thickness. Furthermore, the nanorod thickness seen that it should be same to each of the sample that nearly to 600 nm.

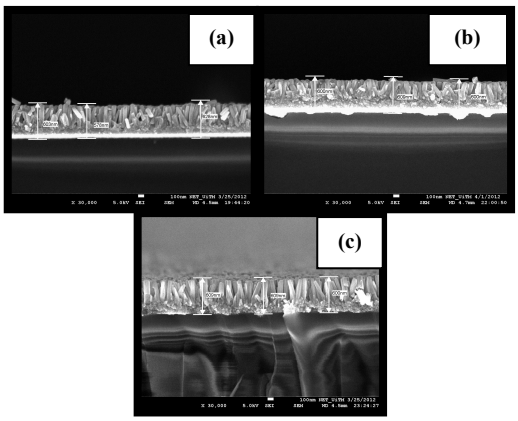

Figure 3(ii): Cross-sectional images of Al doped ZnO nanorod arrays annealed in different environment: (a)oxygen [1 L/min], (b) air, and (c) vacuum.

Figure 4 (a) show the optical properties for transmittance spectra was analyze using UV-vis-NIR spectrophotometry of Al doped ZnO nanorod arrays of seed-layer, as-grown and oxygen annealing with different flow rate such as 0.2 L/min, 0.4 L/min, 0.6 L/min, 0.8 L/min, and 1.0 L/min. Meanwhile for figure 4 (b) is the transmittance spectra of Al doped ZnO nanorod arrays annealed in different environment such as oxygen (1 L/min), air, and vacuum. Generally for the transmittance spectra through the wavelength range set from 300 to 1400 nm show that the nanorod arrays performance a good transmittance spectra in visible region (400-800 nm) where the nanorod arrays are uniformly deposited each of the substrate but it has seen to be decreased in wavelength below 400 nm.

Figure 5 (a) show the absorbance spectra of Al doped ZnO nanorod arrays of seed-layer, as-grown and oxygen annealing with different flow rate such as 0.2 L/min, 0.4 L/min, 0.6 L/min, 0.8 L/min, and 1.0 L/min. It should be observed that absorption spectra show that the wavelength below 400 nm seen improved rather than seed layer that below 2% but between 2 to

4% of absorbance is for the as-grown and nanorod arrays annealed in oxygen with different flow rate which oxygen

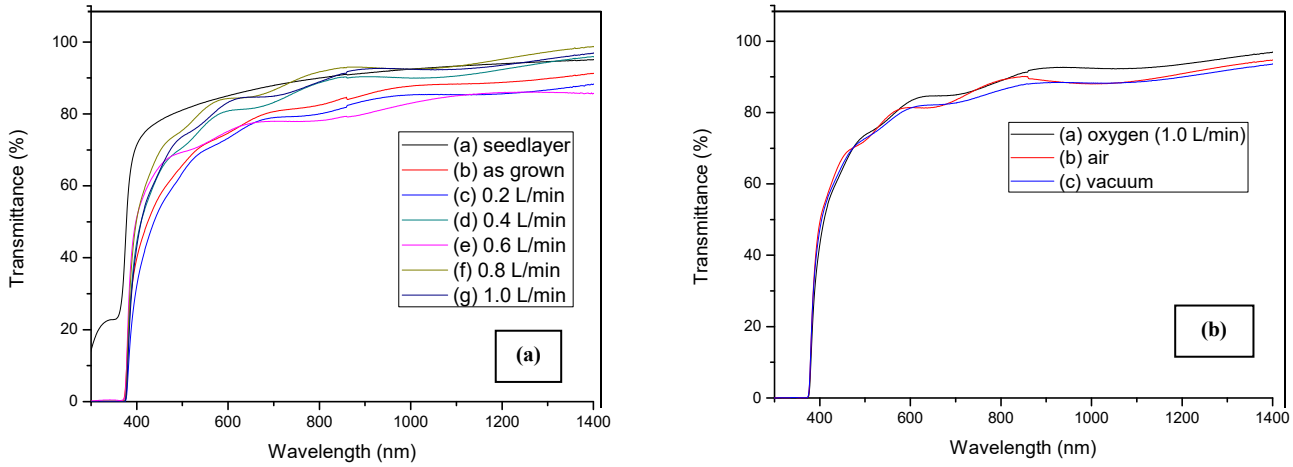

Figure 4: (a) transmittance spectra of Al doped ZnO nanorod arrays of seed-layer, as-grown and oxygen annealing with different flow rate, (b) transmittance spectra of Al doped ZnO nanorod arrays annealed in different environment.

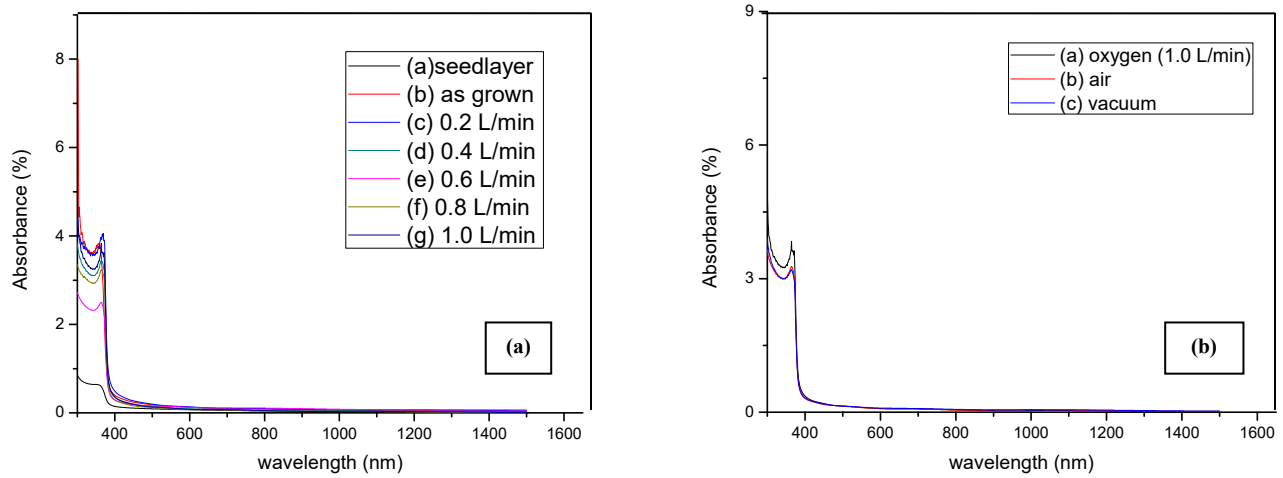

Figure 5: (a) absorbance spectra of Al doped ZnO nanorod arrays of seed-layer, as-grown and oxygen annealing with different flow rate, (b) absorbance spectra of Al doped ZnO nanorod arrays annealed in different environment.

ambient attract in good UV absorption with an absorption edge of 380 nm that related to the direct transition of the electrons between the edge of valence band and conduction band and the wavelength (>400 nm) show the curve become smooth that almost 0% of absorbance. It is also seen at figure 5 (b) which the absorbance spectra of Al doped ZnO nanorod arrays annealed in different environment such as oxygen (1 L/min), air, and vacuum give same properties which the UV absorption is in between 3 to 5% of absorbance that also classified as good absorbance spectra.

Figure 5 (a) and (b) shows the I-V curve is in ohmic behavioral. For the figure 5 (a) is Al doped ZnO nanorod arrays of as-grown and oxygen annealed with different flow rate such as 0.2 L/min, 0.4 L/min, 0.6 L/min, 0.8 L/min, and 1.0 L/min. The result can be observed due to the value of current respect to

the voltage. The resistance should be measure due to formula V=IR, so the Al doped ZnO nanorod arrays of as grown and annealed in oxygen with different flow rate are calculated as resistance result for the figure 5(a) is for as-grown at point

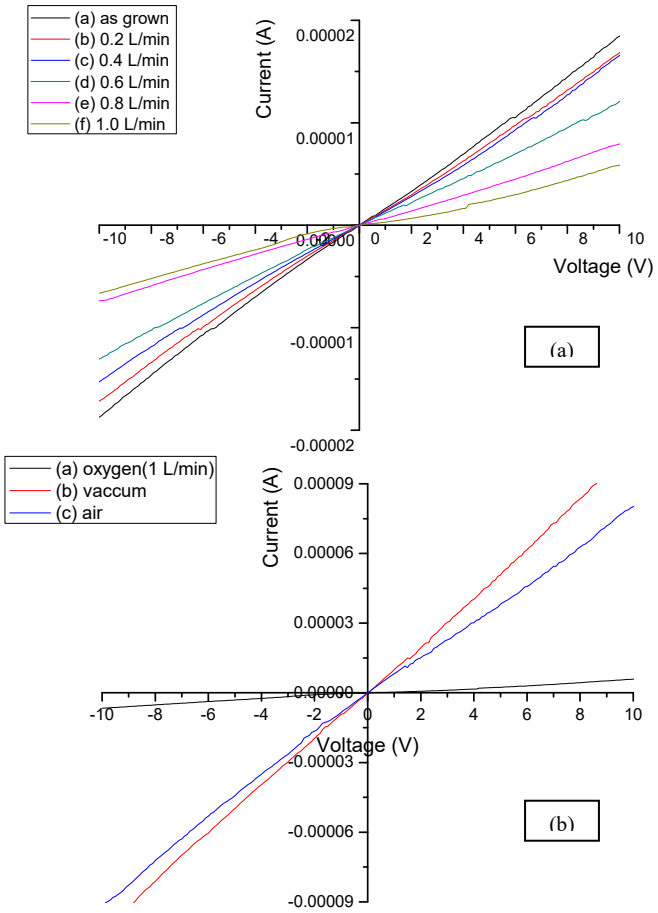

Figure 5: I-V characteristic: (a) Al doped ZnO nanorod arrays of as-grown and oxygen annealed with different flow rate, (b) Al doped ZnO nanorod arrays annealed in different environment.

at point (6V,0.0075mA) and (4V,0.00625mA) is 0.96MΩ, 0.8 L/min at point (8V,0.00625mA) and (4V,0.0025mA) is 1.07MΩ, and lastly 1.0 L/min at point (8V,0.00375mA) and (5V,0.00125mA) is 1.2MΩ.. It means increasing flow rate of oxygen from 0.2 L/min till 1.0 L/min the resistance becomehigher due to voltage versus current. But for figure 5(b) is Al doped ZnO nanorod arrays annealed in different environment show that the value of resistance that measure is for the oxygen (1 L/min) at same with previous resistance which is 1.2MΩ, vacuum at point (7V,0.00525mA) and (4V,0.03mA) is 0.09MΩ and follow by air at point (8V,0.00375mA) and (5V,0.0196mA) is 0.13MΩ. All the measurement take under the (8V,0.015mA) and (4V,0.0075mA) is 0.53MΩ, 0.2 L/min at point (8V,0.0125mA) and (3V,0.005mA) is 0.67MΩ, 0.4 L/min at point (6V,0.01mA) and (4V,0.005mA) is 0.8MΩ, 0.6 L/min

light as UV photoconductive sensor and dark as UV illumination.

Last but not least observed in figure 6 (a) and (b) that show the photoresponse spectra of Al doped ZnO nanorod arrays based UV sensors that annealed at different oxygen flow rate and annealed in different environment with a bias voltage of 10V. From the graph show that the reaction are happen when the UV light are illuminated by 365 nm and expose to the substrate that consist of power density of 750 uW/cm^2, the current increasing sharply due to the ability of substrate react with the UV light if the UV light are ON until the reaction between free electron to the surface of the substrate become limit at one stage and remain steady illumination. But when the UV light are OFF of the current seem become decreasing with fast and in certain period become slowly and until it reach to the initial value, means the UV light that illuminated by 365 nm has enough criteria for detection each the sensitivity performance of fabricated sensor.

The result also show that at as-grown is the lowest of photocurrent because at this level has been seen that it not stable that not too sensitive with UV light which give the dark current is 7.98 x10^{-6} and photocurrent is 3.92 x10^{-5} and the ratio is 4.91. When the substrate annealed with oxygen with different rate it become increasing of the sensitivity which at 0.2 L/min, 0.4 L/min, 0.6 L/min, 0.8 L/min, and 1.0 L/min give a dark current/photocurrent value is 3.70 x10^{-6}/5.59 x10^{-5}, 3.85 x10^{-6}/8.04 x10^{-5}, 3.49 x10^{-6}/1.53 x10^{-4}, 4.63 x10^{-6}/1.56 x10^{-4}, and 3.16 x10^{-6}/1.69 x10^{-4} with the ratio 15.11, 20.8, 43.8, 45.4, and 53.48, the ratio value calculated with divided the photocurrent with dark current which the value of ratio also mean the sensitivity of the substrate with UV light. Meanwhile for the Al doped ZnO nanorod arrays annealed in different environment show that the vacuum have the highest photocurrent but low dark current, same with the air but for oxygen is same with the rate of 1 L/min which for vacuum, air and oxygen dark/photocurrent is 5.53 x10^{-5}/2.45 x10^{-4}, 2.38 x10^{-5}/1.81 x10^{-4}, and 3.16 x10^{-6}/1.69 x10^{-4} with the ratio is 4.43,7.61 and 53.48. The photoresponse characteristic of sensors fabricated was observed for Al doped ZnO nanorod arrays which measure with bias voltage of 10 V under 365 nm UV illumination light (750 uW/cm^2). From the table show that the calculation for reponsivity, R can be apply such as:

$$R = \frac{Iph - Idark}{Pop}$$

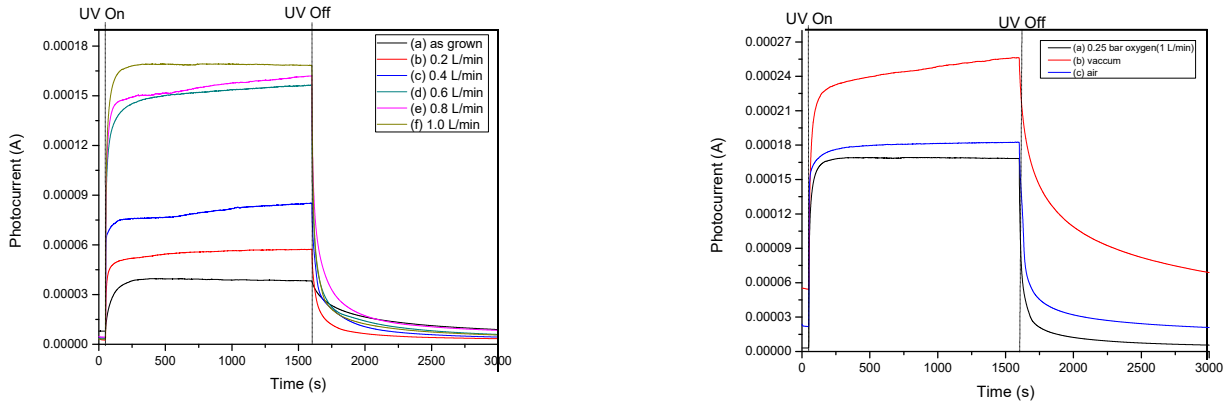

Figure 6: Photocurrent properties of (a) Al doped ZnO nanorod arrays of as-grown and oxygen annealed with different flow rate, (b) Al doped ZnO nanorod arrays annealed in different environment.

Table 1: Characteristic of Al doped ZnO nanorod arrays of as-grown and oxygen annealed with different flow rate under 365 nm UV illumination with an optical power of 750uW/cm^2.

Sample substrate	Dark current (A)	Photocurrent (A)	Sensitivity /Ratio	Responsivity (A/W)
as grown	7.98×10^{-6}	3.92×10^{-5}	4.91	0.67
0.2 L/min	3.70×10^{-6}	5.59×10^{-5}	15.11	1.16
0.4 L/min	3.85×10^{-6}	8.04×10^{-5}	20.8	1.7
0.6 L/min	3.49×10^{-6}	1.53×10^{-4}	43.8	3.32
0.8 L/min	4.63×10^{-6}	1.56×10^{-4}	45.4	3.11
1.0 L/min	3.16×10^{-6}	1.69×10^{-4}	53.48	3.69

Where I_{ph} is the photocurrent, I_{dark} is the current and Pop is the optical power of the UV source. So it is accepted that when the UV illumination is turned on and off seem that the photoresponse of UV photoconductive sensor involve by the adsorption and desorption of the oxygen on the surface. The absorbed occur when oxygen from surrounding area react to the surface of the substrate by capture the ZnO according to the following reaction:

Table 2: Characteristic of Al doped ZnO nanorod arrays annealed in different environment under 365 nm UV illumination with an optical power of 750 uW/cm^2.

Sample substrate	Dark current (A)	Photocurrent (A)	Sensitivity/ Ratio	Responsivity (A/W)
oxygen (1 L/min)	3.16×10^{-6}	1.69×10^{-4}	53.48	3.69
air	2.38×10^{-5}	1.81×10^{-4}	7.61	3.97
vaccum	5.53×10^{-5}	2.45×10^{-4}	4.43	5.32

$$O_2 + e^- \rightarrow O_2^- \quad (1)$$

where the O_2 is oxygen molecule, e^- is the free electron, and O_2^- is the adsorbed oxygen on the nanorod surface which if the UV light turn on it react and electron-hole pair s are photogenerated due to these reacton:

$$h\nu \rightarrow h^+ + e^- \quad (2)$$

which hv is the photon energy of UV light, h^+ is the photogenerated hole in valence band and e^- is the photogenerated electron at the conduction band. The reaction of photogenerated hole that react with the negative charged

adsorbed oxygen to release oxygen molecules from nanorod surface. These are the reaction due to current condition:

$$O_2^- + h^+ \rightarrow O_2 \qquad (3)$$

If these condition occurs, the electron of the electron-hole pair at the conduction band should be remove to the outer space. But the oxygen molecule recombine with electron in the turned off condition of UV illumination that can be decrease of the film conductivity back to the initial value or dark current.

IV. CONCLUSION

The metal oxide semiconductor material through UV photoconductive sensor were successfully fabricated of Al doped ZnO nanorod arrays prepared using sonicated sol-gel immersion that anneal in different environment and different oxygen flow rate was successfully done. The annealing process for each of ambient condition such as air, oxygen and vaccum suppose improved the crystallinity of the nanorod arrays. From the FESEM result show that the thickness of the substrate seem equal to each other which is nearly to 600 nm and the top surface has diameter is in between 40-60 nm. The optical transmittance and absorbance spectra show the absorption of UV light is good at 400 to 800 nm as the visible region. The nanoholes occur at the surface of the substrate due to annealing process and it seem much more occur at the surface of substrate anneal in air ambient rather than oxygen annealed and vacuum annealed. From the I-V result also has been suggest that oxygen annealed with more flow rate give high resistance which is 1 L/min rather than vacuum annealed or air annealed. So annealed in oxygen has give more sensitivity that expose with UV photoconductive sensor.

V. ACKNOWLEDGEMENT

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