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Article

Efficiency Improvement on Indium Tin Oxide Films for Dye-Sensitized Solar Cell using Oxygen Plasma by Bias-Magnetron RF Sputtering Process

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Abstract: DSSC devices are among the most widely studied thin-film solar cells because of their cost-effectiveness, low toxicity, and simple fabrication method. However, there is still much scope for replacing current DSSC materials due to their high cost, low volume, and lack of long-term stability. Accordingly, ITO-nanorod films were fabricated by E-beam evaporation using the glancing angle deposition method in this study. Then, the ITO-nanorod was treated by oxygen plasma via a bias-magnetron RF sputtering process to improve the efficiency of DSSC under a varying gas flow rate of 20, 40, 60, 80, and 100 sccm. The FE-SEM investigation of the ITO film structure revealed that the obtained nanorod structures have slightly different diameters. At the same time, an increase in the oxygen flow rate resulted in a rougher film surface structure. In this, the lower sheet resistance was received because of rougher morphology. When comparing the DSSC efficiency test results, we found that at a gas flow rate of 100 sccm, the highest efficiency value showed by 9.5%. On the other hand, the ITO-nanorod without plasma treatment exhibited the lowest efficiency. Hence, plasma technology can be practically applied to improve the efficiency of DSSC devices. This study will be a prototype of a highly advanced solar cell manufacturing method for the solar cell industry.

Keywords: ITO-nanorod; oxygen plasma; RF sputtering; DC-self bias; DSSCs

1. Introduction

Recently, dye-sensitized solar cells (DSSCs) have gained much attention in solar energy studies because they are high-efficiency materials and can be easily fabricated compared to other solar cells, such as silicon [1,2]. In addition, these solar cells are feasible to produce at low cost. Titanium dioxide (TiO₂) photoelectrode plays an essential role in DSSC devices because it enables DSSC to function, which needs the dye molecules to be attached to the TiO₂ particle surface. Here, the molecules absorb sunlight, and electrons from the primary state will be excited to a high level, from where they pass into the conduction band of TiO₂. Electrons permeate through the TiO₂ nanoporous structure to the transparent conducting film, flowing through the external load to the counter electrode. Therefore, the performance of DSSCs depends on many factors, such as the film's structure, morphology, and thickness, the type of dye and electrolyte, and the electrical resistance of the electrodes [3,4]. The film surface must be porous, the adhesion to the surface must be good, and the TiO₂ particles must be well interconnected. The particles should also be designed in nano-sized [5,6]. Significantly, the key factors

influencing the performance of DSSC devices are the transparent surface characteristics and electrical resistance. In DSSC devices, there are two types of transparent conductive oxides (TCOs), including fluorine-doped tin oxide (FTO) and indium-doped tin oxide (ITO). Although FTO is currently the most widely used, it has several disadvantages, like rough surface and lower transparency. Meanwhile, ITO has lower sheet resistance, better stability and adhesion, can be fabricated on a larger surface, and has a lower processing temperature than FTO. ITO materials are generally n-type semiconductors with an optical bandgap greater than 3.4 eV [7,8]. Several research groups have previously reported and suggested that modifying the surface properties of ITO is an essential step for the fabrication of solar cell devices [9–13]. Various methods have been developed for preparing ITO surfaces, including ultraviolet-ozone treatment [14–16], wet treatment annealing process [17,18], and gas plasma [19,20]. Among these, oxygen plasma is a promising therapy because of its many advantages, outstanding functionality, smoothest surface, and lowest sheet resistance [21–23]. However, improving the ITO surface with a technique conducive to depositing a good-quality film is necessary. A bias applied to the surface can attract positive ions in the plasma to attack the grown film [24–26]. This bombardment causes the molecules and clusters to condense on the surface to gain energy, and the molecules detach from the film with weak bonds [27]. This type of bombardment is beneficial to the crystallinity of the film, and the effect of suitable ions on the deposited film improves the film's properties [28–30].

In this study deposited the ITO films on glass substrates by E-beam evaporation with glancing angle deposition. Then, the ITO was deposited on glass treated by oxygen plasma with a bias-magnetron RF sputtering process. The effects of oxygen gas content on the film surfaces and electrical, optical, and efficiency of DSSC devices were evaluated.

2. Materials and Methods

2.1. ITO Film Fabrication

To prepare the ITO films, the evaporation material used indium tin oxide pieces, which had a compound of indium oxide (In_2O_3) with tin (SnO_2) at 99.99% purity, produced by Kurt J. Lesker Company, United States. The samples used glass substrates produced by As One Corporation, Osaka, Japan. The film depositing machine used a DVB-SJC model manufactured by Denton Vacuum Inc., United States. The electron beam generator used a TFI-270-01 model of 4 crucibles manufactured by Denton company, England. During the deposition process, argon gas (Ar) of 13 sccm and oxygen gas (O_2) of 8 sccm were used. The pressure in a chamber was pumped down to 2.6×10^{-5} Torr at room temperature.

2.2. Oxygen Plasma Treatment with Bias-Magnetron RF Sputtering Process

The next step is to treat the ITO films using a bias-magnetron RF sputtering process. The equipment applied with the sputtering machine used ATC 2000-F, manufactured by AJA International, Inc., United States, as shown in Figure 1. During the oxygen plasma treatment process, argon gas (20 sccm) is used as an inert gas. The substrate bias can be adjusted from -100 to +100 V, and only the oxygen gas ratio was varied by 20 sccm, 40 sccm, 60 sccm, 80 sccm, and 100 sccm, in order to examine the effect of oxygen plasma on the composition changes of the ITO films. The pressure inside the chamber was pumped to 2.6×10^{-2} Torr.

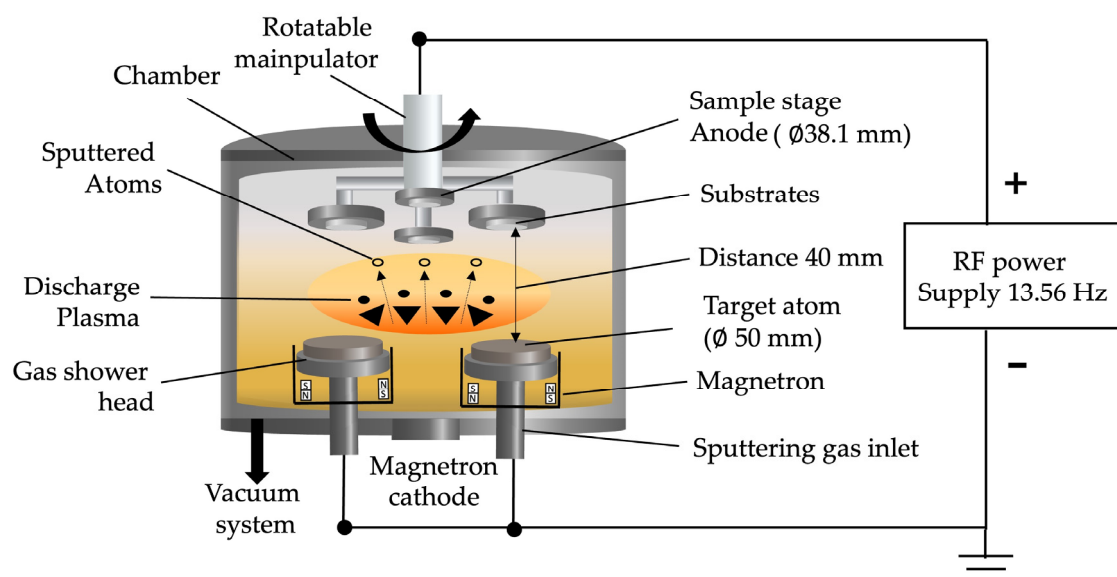


Figure 1. Schematic diagram showing equipment of deposition machine applied with plasma treatment process with bias-magnetron RF sputtering of varying gas at contents 20 sccm, 40 sccm, 60 sccm, 80 sccm, and 100 sccm.

2.3. Dye-Sensitized Solar Cell Preparation

In this work, ITO was cut into 2 x 3 cm sizes to investigate the efficiency of solar cells. The fabrication of DSSC devices was done by a screen-printing technique [31]. At the beginning of the process, the prepared devices were put into an oven at 60 C for 1 hour, then burned at 450 C for 2 hours. N719 dye is used as the photosensitizer material manufactured by Ossila Ltd., United Kingdom. N719 dye was injected into the cells and covered with parafilm to prevent evaporation. The cells were then left for 48 hours. After that, the cells were assembled and tested for efficiency.

2.4. Characterization Method

The diffraction pattern was tested and compared with the JCPDS standard values using an X-ray diffractometer (XRD) of the TTRAI model manufactured by Rigaku company, Japan. Moreover, inspection was performed using the grazing incidence X-ray diffraction (GIXRD) method. Field emission scanning electron microscope: FE-SEM of the S4700 model, manufactured by Hitachi company, Japan, was used to examine the thickness of thin films in a cross-sectional imaging system and to determine the grain size in the surface imaging system at different depositing conditions. UV-vis spectrophotometer model Cary 7000 UMS (Agilent Technologies company, United States) was used to analyze the optical properties of ITO film within the measurement range of 200-2200 nm, displaying the percentage transmittance (%T). The point probe model RM3 (Jandel company, United Kingdom) was used to measure electrical properties with the Hall effect model HMS-3000 (Ecopia Corporation, Republic of Korea). In addition, solar simulator model XPS-300 (Solar light company, Inc., United States) was used to test the efficiency of DSSC devices under the short circuit current or current density (J_{sc}), open circuit voltage (V_{oc}), maximum current (I_{max}), maximum power (P_{max}) and fill factor (FF).

3. Result

The characterization of ITO films prepared by E-beam Evaporation with glancing angle deposition was performed via using the sputter etching process under oxygen plasma treatment with different gas supplies of 20 sccm, 40 sccm, 60 sccm, 80 sccm, and 100 sccm. During the sputter etching process on ITO film with oxygen plasma discharge, the reactor chamber is filled with a weakly ionized plasma, which consists of an ionized gas containing electron (e^-), positively charged ions (O^+ , O^{2+} , Ar^+), negatively charged ions (O^-), and a large number of neutral species such as and atoms.

The species diffusing to the surface causes crystal changes. In this case, when the plasma is turned off while oxygen gas is introduced into the reactor chamber at a constant rate, the analysis of the plasma diagnosis in the form of that is determined by the following reactions [32–34].



Figures 2 and 3 shows the surface mechanism of oxygen plasma treated on ITO-nanorod substrates by bias-magnetron RF sputtering process of the varying gas flow rate of 20, 40, 60, 80, and 100 sccm. During the RF sputtering process, the RF power generator changes the target electrode, attracting electrons and ions alternately. For this, electrons are lighter than ions and have a higher mobility. Thus, they collide with the target more often, resulting in excess negative charge and a direct electric field formation on the target [35].

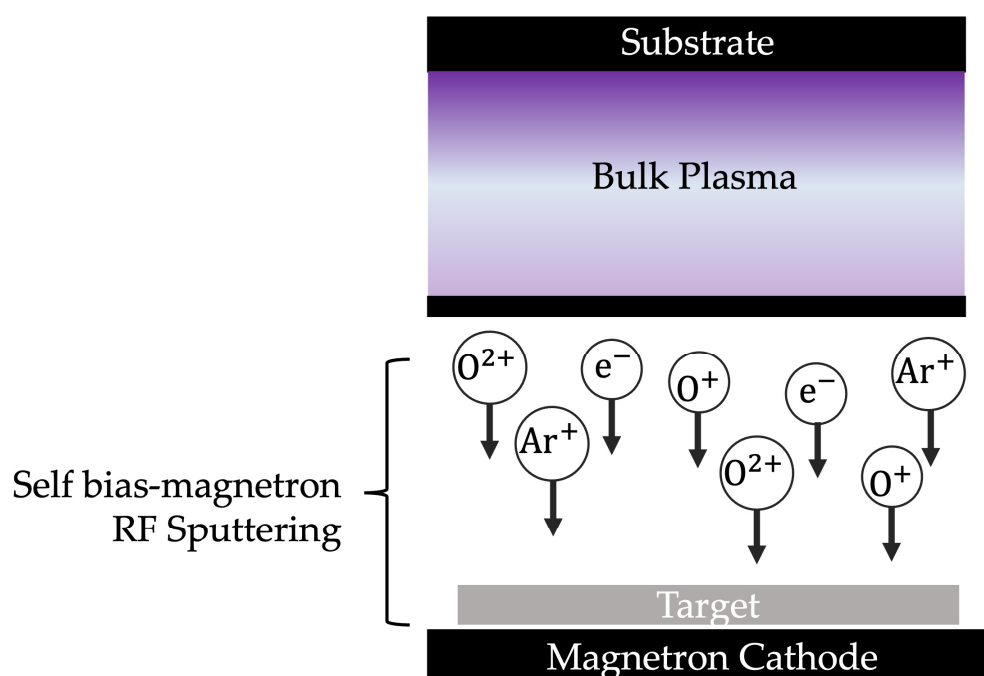


Figure 2. Bias-magnetron RF sputtering mechanism used for oxygen plasma treatment on ITO-nanorod film under different oxygen gas flow rates of 20, 40, 60, 80, 100 sccm.

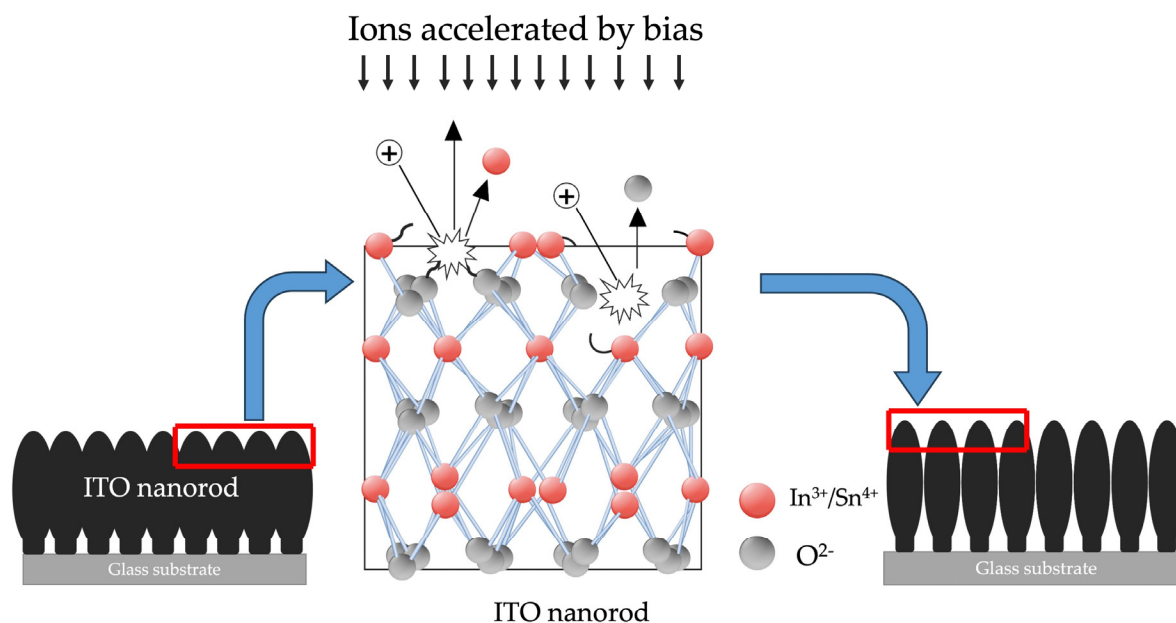


Figure 3. Processing of oxygen plasma bombardment to the ITO-nanorod surfaces driving under bias-magnetron RF sputtering method with different gas conditions of 20, 40, 60, 80, and 100 sccm.

3.1. ITO Film Structure

Figure 4 illustrates the X-ray diffraction patterns of ITO film before oxygen plasma treatment (As-deposited: ASD) and after oxygen plasma treatment at a flow rate of 20, 60, 80, and 100 sccm as can be seen in the figure; the X-ray diffraction patterns of the ITO surface show diffraction planes at (222) and (440) with 2θ of 30.7 and 51.3, respectively. When considering the intensity of the peaks from all apparent planes (222 and 440), it was found that when the oxygen gas flow rate increased, the crystal structure after plasma treatment changed slightly.

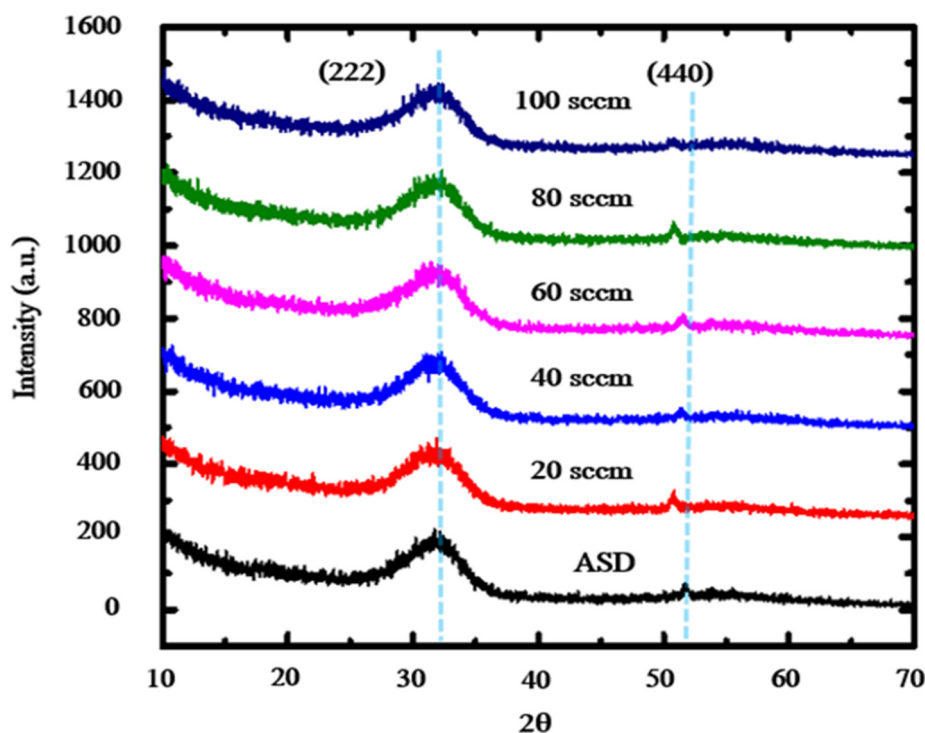


Figure 4. X-ray diffraction patterns of ITO films preparing with E-beam evaporation with glancing angle deposition and treated by oxygen plasma of varying oxygen gas, such as 20, 40, 60, 80, and 100 sccm.

The surface analysis of ITO films grown on plasma treatment under different oxygen gas supplies at a flow rate of 20, 40, 60, 80, and 100 sccm was performed using the scanning electron microscopy analysis method. The results of cross-sectional photographs are shown in Figure 5. The photographs show the structure of the films grown on the substrate as nanorods or ITO-nanorods, relating to Figure 3. Similarly, Figure 6 displays the photograph characteristics of the top film surface. The figure shows the apparent changes in the ITO-nanorod surface treated with oxygen plasma. When the oxygen gas flow rate increases to 100 sccm, the crystal size becomes smaller and separated compared to a lower oxygen gas flow rate. The result is due to the influence of plasma etching on the ITO-nanorod surface, which results in breaking ITO bonds and changing the top film layer.

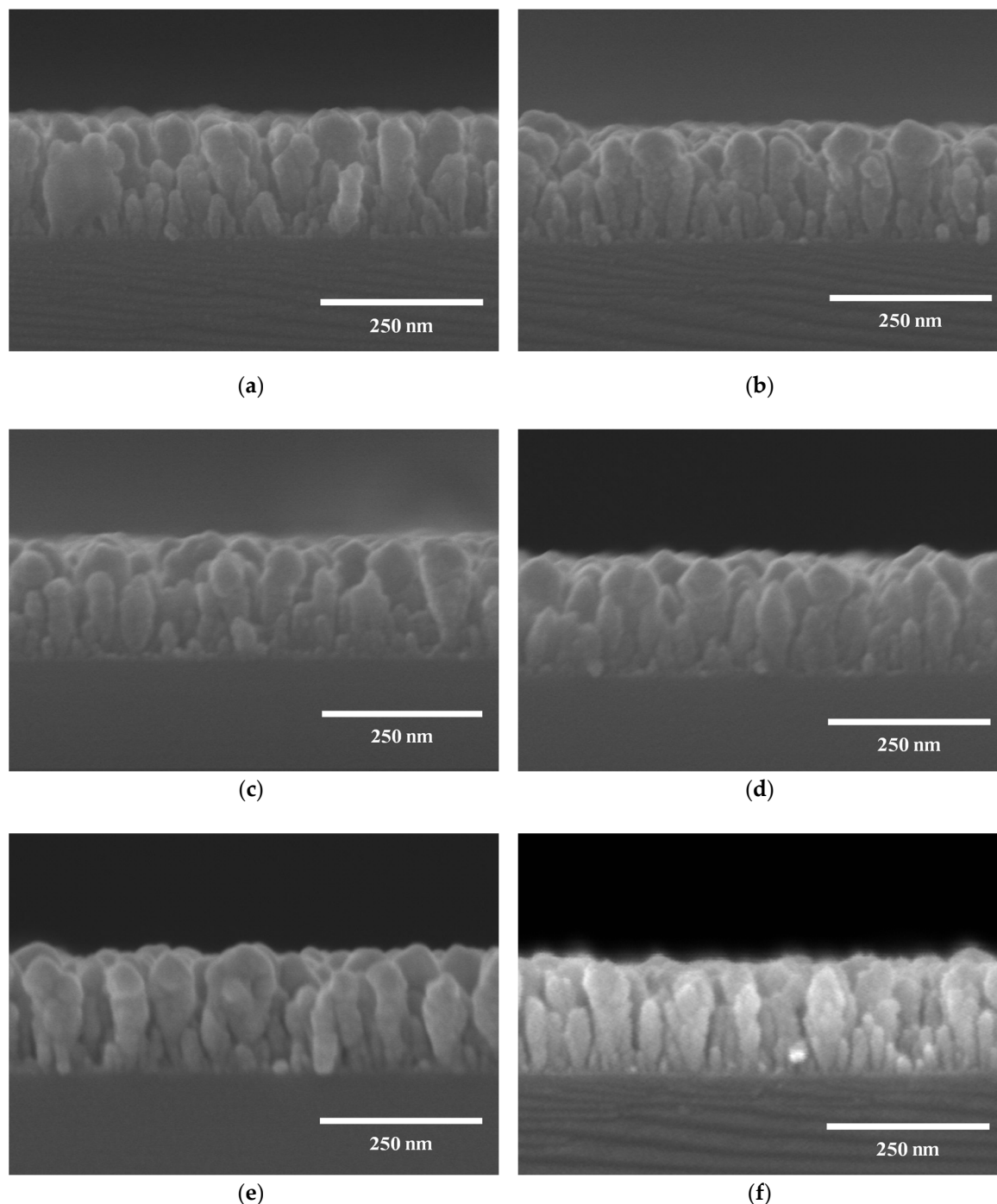


Figure 5. Cross-section images of scanning electron microscopy analysis on ITO-nanorod deposited on glass substrate treated by oxygen plasma of varying gas flow rate comparing with as-deposited film: (a) as-deposited; (b) 20 sccm; (c) 40 sccm; (d) 60 sccm; (e) 80 sccm; (f) 100 sccm.

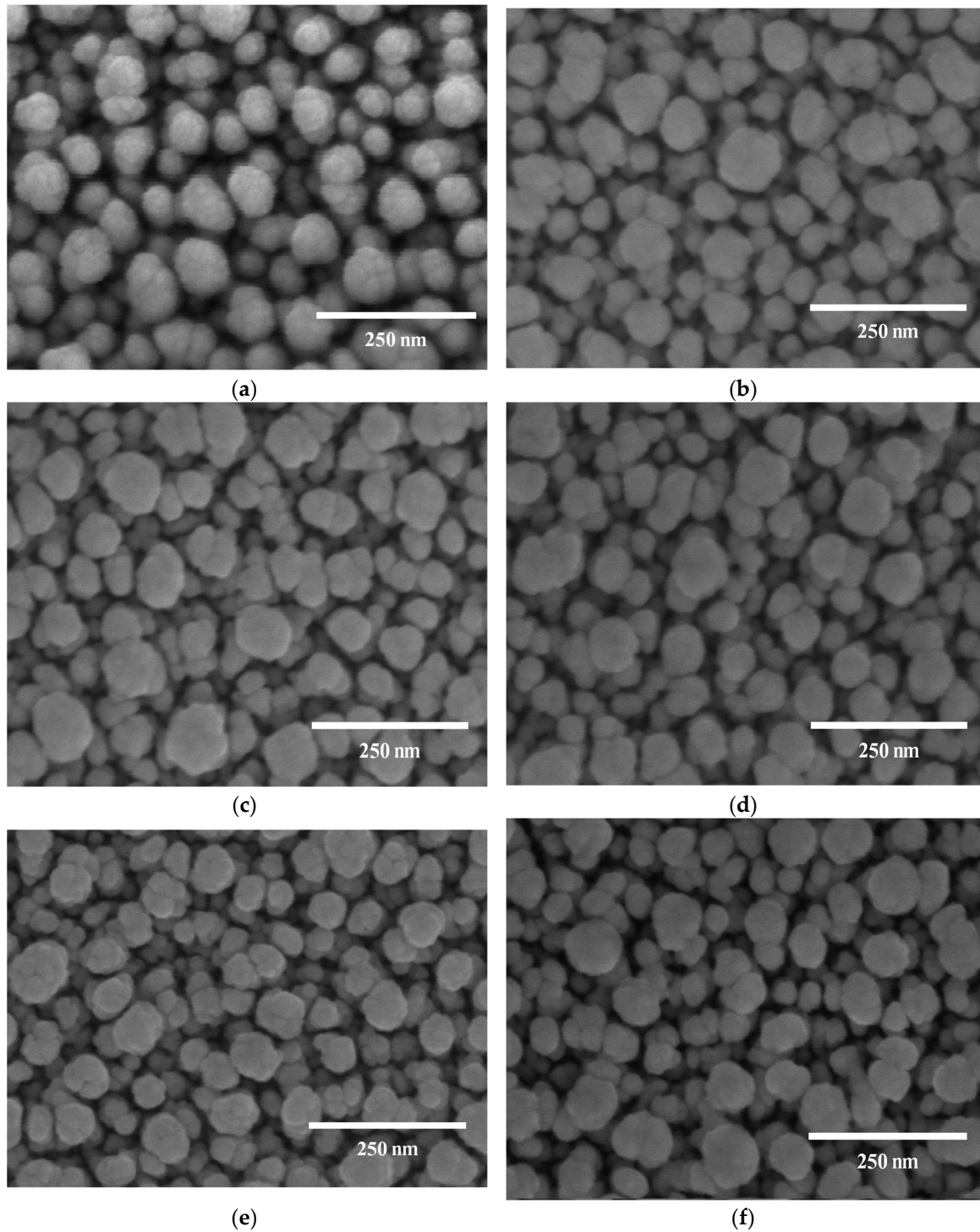


Figure 6. Top-image of scanning electron microscopy analysis (top view) of on ITO-nanorod deposited on glass substrate treated by oxygen plasma of varying gas flow rate comparing with as-deposited film: (a) as-deposited; (b) 20 sccm; (c) 40 sccm; (d) 60 sccm; (e) 80 sccm; (f) 100 sccm.

Additionally, the diameter and length of ITO-nanorod structures treated with oxygen plasma at different flow rates were analyzed, and the average values were calculated using Gaussian distribution [36] according to equation 5.

$$f_g(X) = \frac{1}{\sqrt{2\pi\sigma^2}} e^{\frac{-(X-a)^2}{2\sigma^2}} \quad (5)$$

Where the Gaussian function value is $f_g(X)$, a is the mean value, the standard deviation is σ , and X is the diameter measurement of each grain in nanometers. Figure 7 illustrates the relationship

graph from the calculation of the diameter and length of ITO-nanorod, which compares the samples between as-deposited film and ITO-nanorod treated with oxygen plasma. The results exhibit that the adjustment of oxygen gas flow rate in the plasma process affects the change in the size of the nanorods, with the length of ITO-nanorods ranging from 199-211 nm. When considering the size of the nanorods in the blue graph at different oxygen gas flow rates, it was found that when the oxygen gas flow rate increased, the nanorod size gradually decreased. The nanorods show the smallest size at a flow rate of 100 sccm compared with other flow rate conditions. Since at a flow rate of 100 sccm, the concentration of oxygen particles is the highest when the plasma is generated, resulting in the ionization of a large amount of oxygen gas. In this case, when stimulated by a biased process, causing more energy to break the bonds on ITO-nanorod surfaces, resulting in smaller nanorod size, the bond-breaking reaction can be seen in Figure 3.

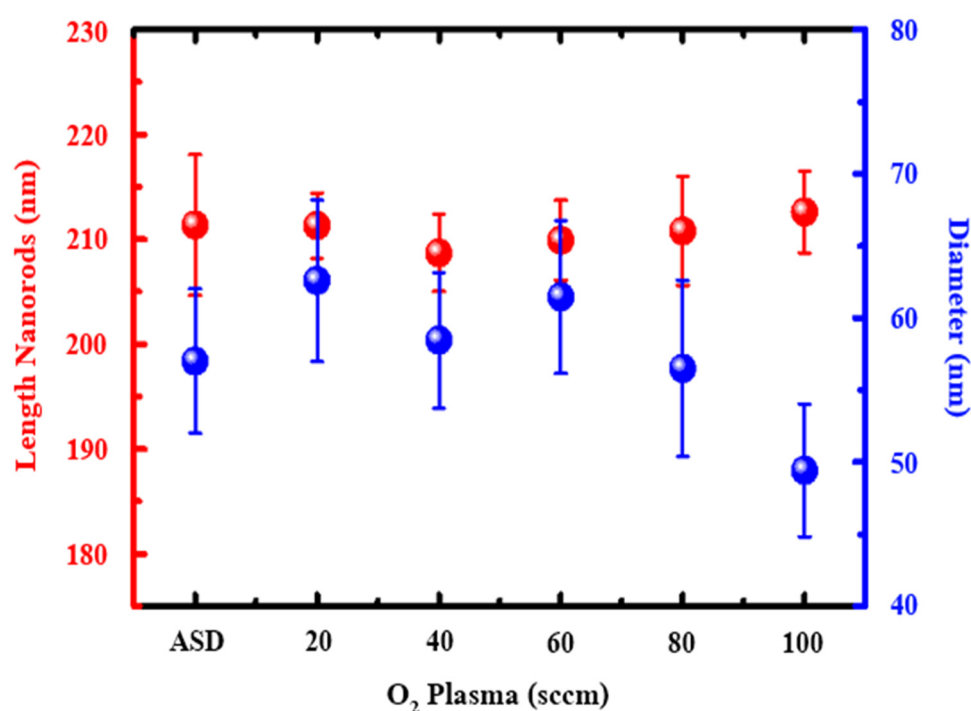


Figure 7. Relationship of length nanorods and diameter size on ITO-nanorod calculated by Gaussian distribution comparing between as-deposited film and oxygen plasma treatment at 20, 40, 60, 80, and 100 sccm.

3.2. Optical Properties

The light transmittance was analyzed by a spectrophotometer measuring from the 250-2000 nm wavelength range. The comparison between glass substrate, as-deposited film, and ITO-nanorod treated with oxygen plasma of varying gas content at 20, 40, 60, 80, and 100 sccm is displayed in Figure 8. From the spectral lines, it was found that the light transmittance of the plasma-treated ITO-nanorod film was in a good range and was close to each other in the visible wavelength range of 380-750 nm, which is the appropriate wavelength. The overall transmittance values are approximately 85%.

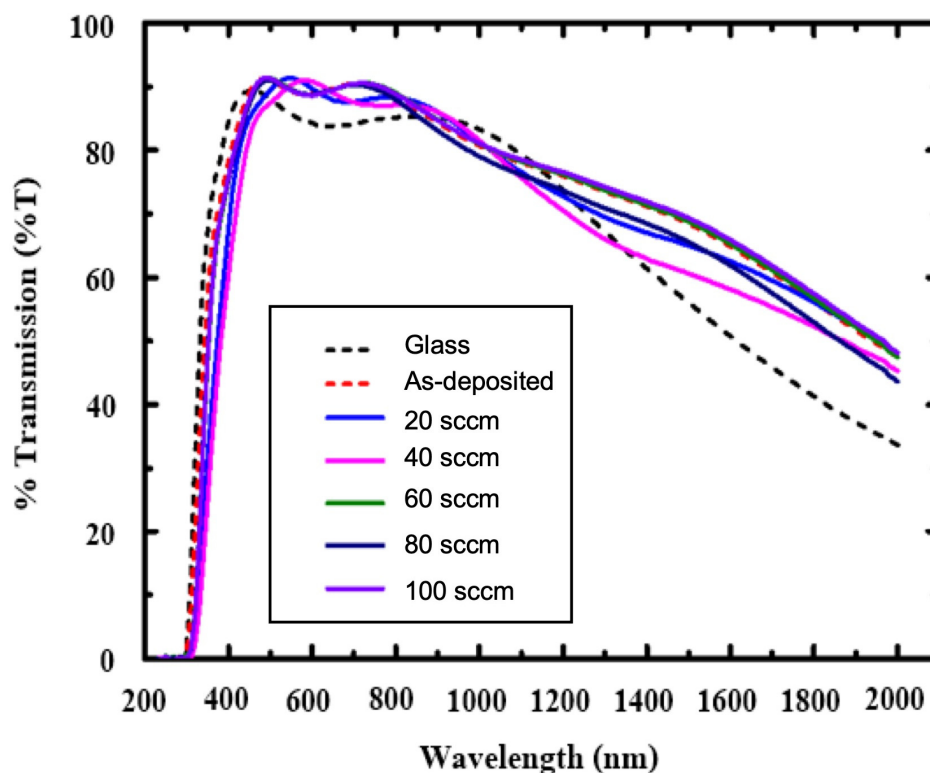


Figure 8. Transmittance spectra of the ITO-nanorod films compared with clear glass, as-deposited film and treated with oxygen plasma content of 20, 40, 60, 80, 100 sccm preparing via bias-magnetron RF sputtering process.

Furthermore, the average transmittance in the visible range (400-700 nm) of ITO-nanorod films treated with oxygen plasma under different gas flow rates of 20, 40, 60, 80, and 100 sccm was calculated and compared with glass, as-deposited film, and ITO commercial, as shown in Figure 9. As can be seen in the graph, the average transmittance in the visible range of the plasma-treated ITO-nanorod film increases with the increase of the oxygen gas flow rate. Due to the increased self-collision of oxygen ions at higher concentrations, a correspondingly higher average transmittance change is achieved. Then, the omnidirectional characteristics were analyzed at 500, 600, and 700 nm wavelength of the glass substrate, as-deposited film, and treated with oxygen plasma of varying gas 20, 40, 60, 80, and 100 sccm. It was measured at the incident angle between 0° and 80° . The analysis results are illustrated in Figure 10–12. From the spectrum, the results found that a percentage of light-transmittance at incident angles of ITO-nanorod treated with oxygen plasma was close in overall cases of gas content. Compared with glass, ITO-nanorod films treated with oxygen plasma have a higher light transmittance. Therefore, optimizing light transmittance value is suitable as a thin transparent conductive oxide film in solar cells.

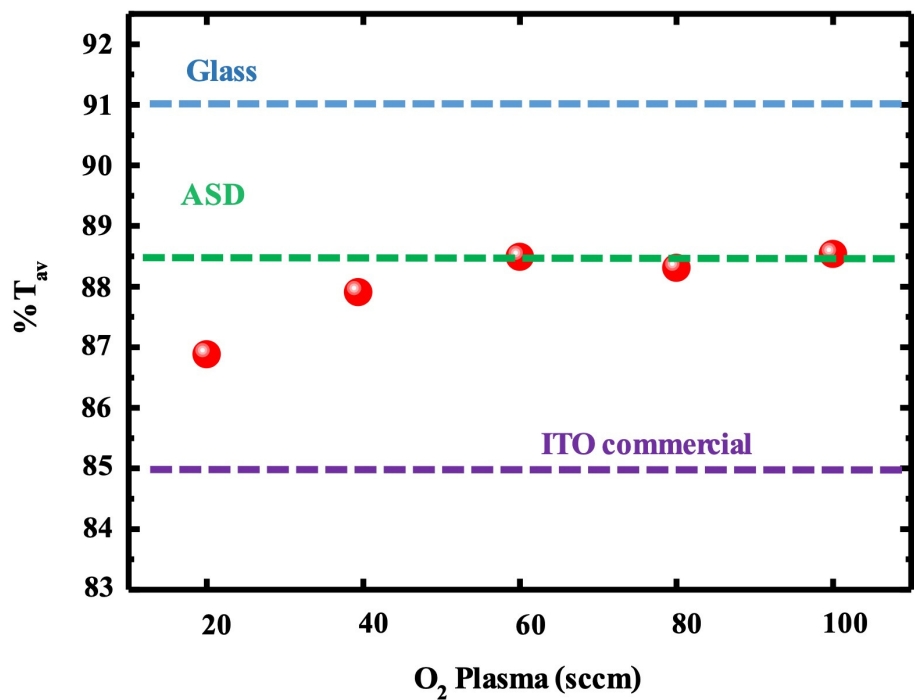


Figure 9. Average transmittance in the visible range of the ITO-nanorod films compared with clear glass, as-deposited film and treated with oxygen plasma content of 20, 40, 60, 80, 100 sccm preparing via bias-magnetron RF sputtering process.

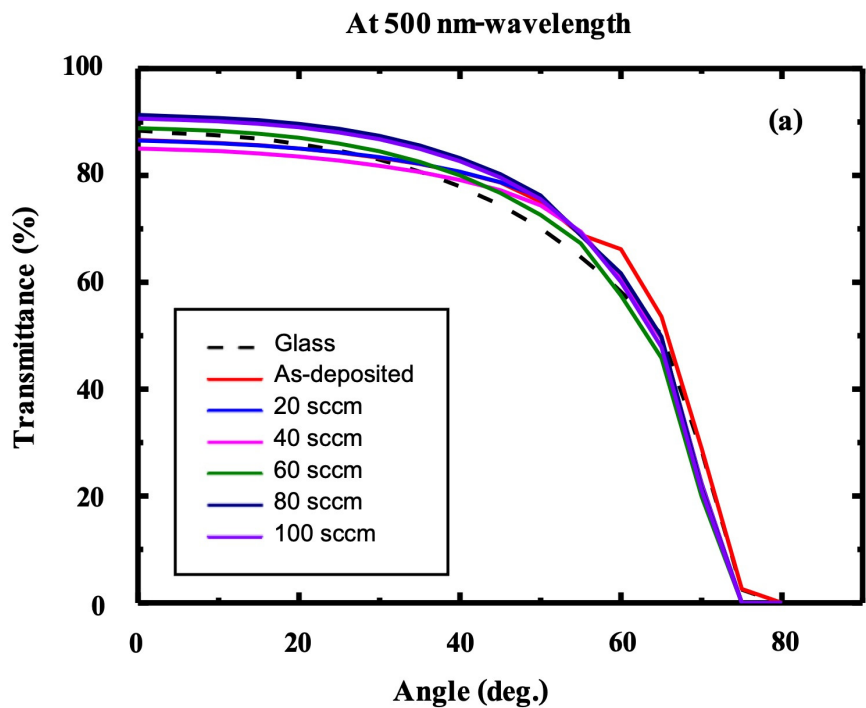


Figure 10. Omnidirectional characteristics on transmittance properties of ITO-nanorod treated with oxygen plasma under gas content 20, 40, 60, 80, 100 sccm comparing to glass substrate and as-deposited film at 500 nm-wavelength.

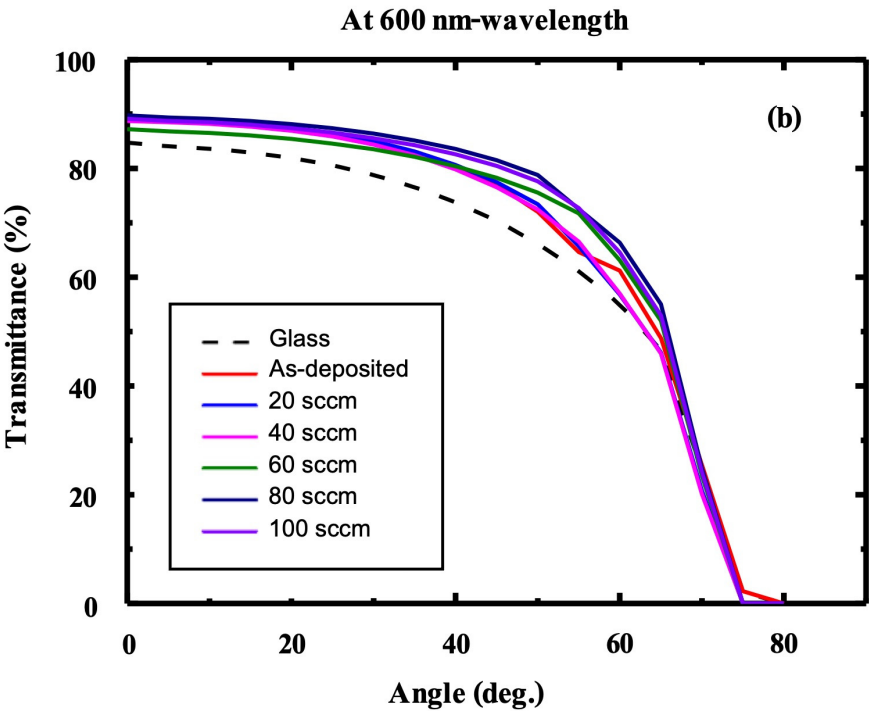


Figure 11. Omnidirectional characteristics on transmittance properties of ITO-nanorod treated with oxygen plasma under gas content 20, 40, 60, 80, 100 sccm comparing to glass substrate and as-deposited film at 600 nm-wavelength.

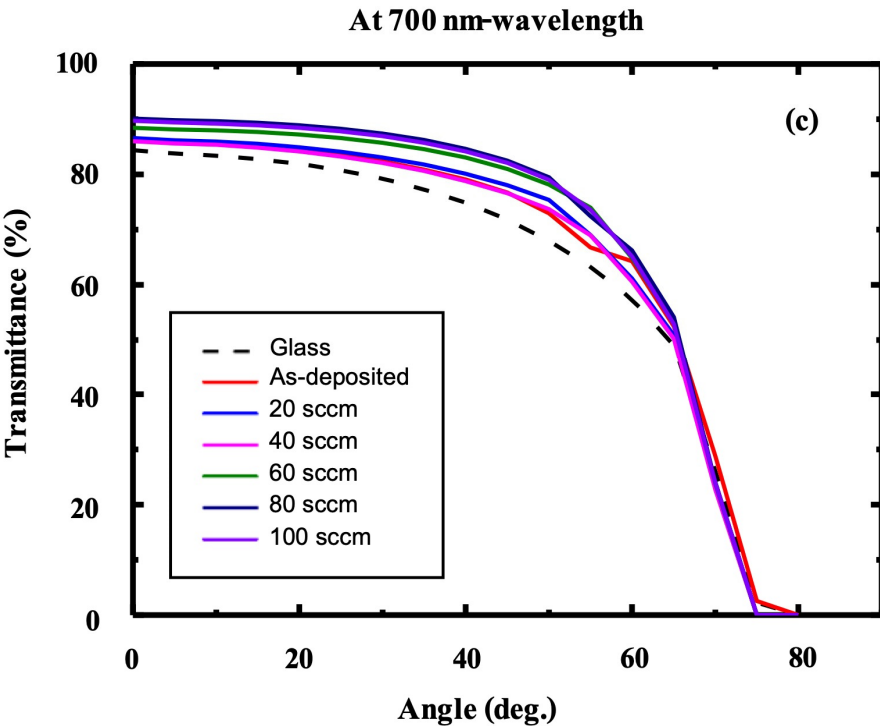


Figure 12. Omnidirectional characteristics on transmittance properties of ITO-nanorod treated with oxygen plasma under gas content 20, 40, 60, 80, 100 sccm comparing to glass substrate and as-deposited film at 700 nm-wavelength.

3.3. Electrical Properties

Figure 13 shows the electrical properties of ITO-nanorod treated with oxygen plasma at gas contents 20, 40, 60, 80, and 100 sccm, compared to as-deposited film. From the electrical properties test results, it was found that when the oxygen gas flow rate increased, the sheet resistance value gradually decreased. It was found that the oxygen gas flow rate of 100 sccm showed the lowest sheet resistance value. On the contrary, the as-deposited film gave the highest sheet resistance value. In this, the decrease in sheet resistance is because of the bombardment of oxygen ions with the film surface with the highest oxygen concentration. Thus, it has a higher ability to attack the ITO-nanorod film surface, which exhibits the lowest sheet resistance. With this low resistance, ITO-nanorod films will greatly benefit DSSCs, as they will enable efficient charge transfer and reduce energy loss in the cells.

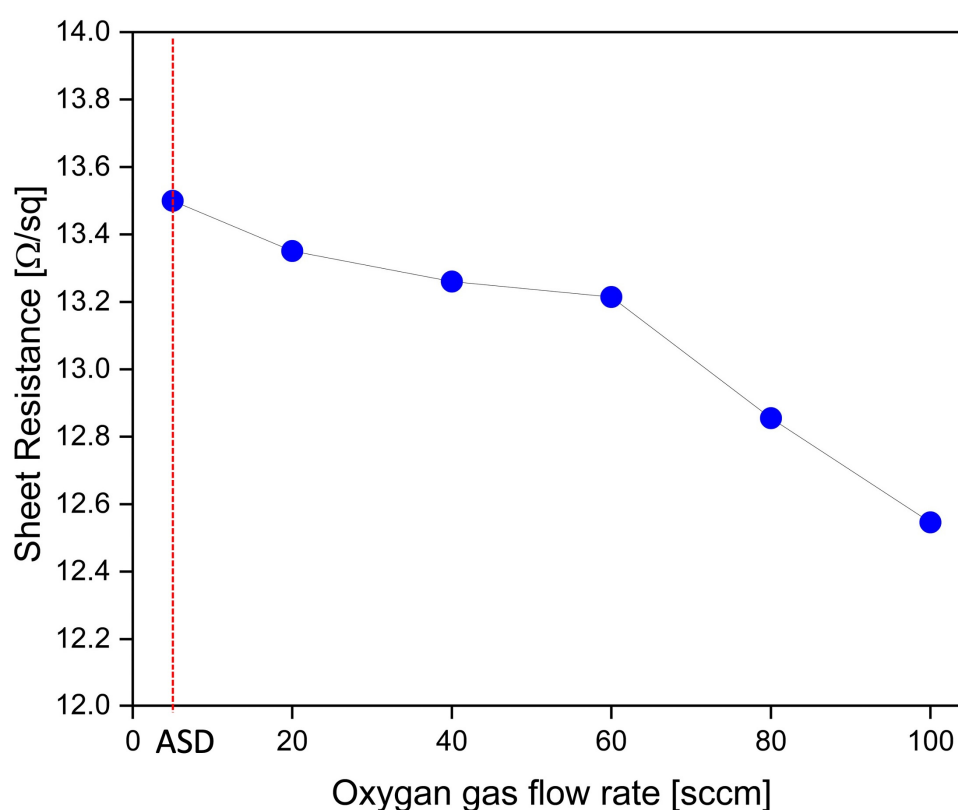


Figure 13. Electrical properties on ITO-nanorod film treated by oxygen plasma comparing with as-deposited film using four point probe technique.

3.4. Efficiency of Dye-Sensitized Solar Cell

In the photovoltaic performance, the DSSC structure is defined as follows: ITO-nanorod treated with oxygen plasma used as a working electrode, deposited with TiO_2 , N 719 dye used as the photosensitizer, I^-/I_3^- used as electrolyte [37], and platinum (Pt) used as the counter electrode. Thus, the assembly structure of the DSSC device is glass-deposited ITO-nanorod/ TiO_2 /(I^-/I_3^-)/Pt, respectively [38–41]. Figure 14 illustrates the I-V characterization on ITO-nanorod film applied with DSSC devices. The overall efficiency (η) of DSSC, meaning the percentage of solar energy that shines on a photovoltaic device, which is converted into electricity, was tested by J_{sc} , V_{oc} , and FF, which are calculated by equation (5) [42], as shown these parameters in Table 1.

$$\eta(\%) = \frac{J_{sc} \times V_{oc} \times FF}{P_{in}} \quad (5)$$

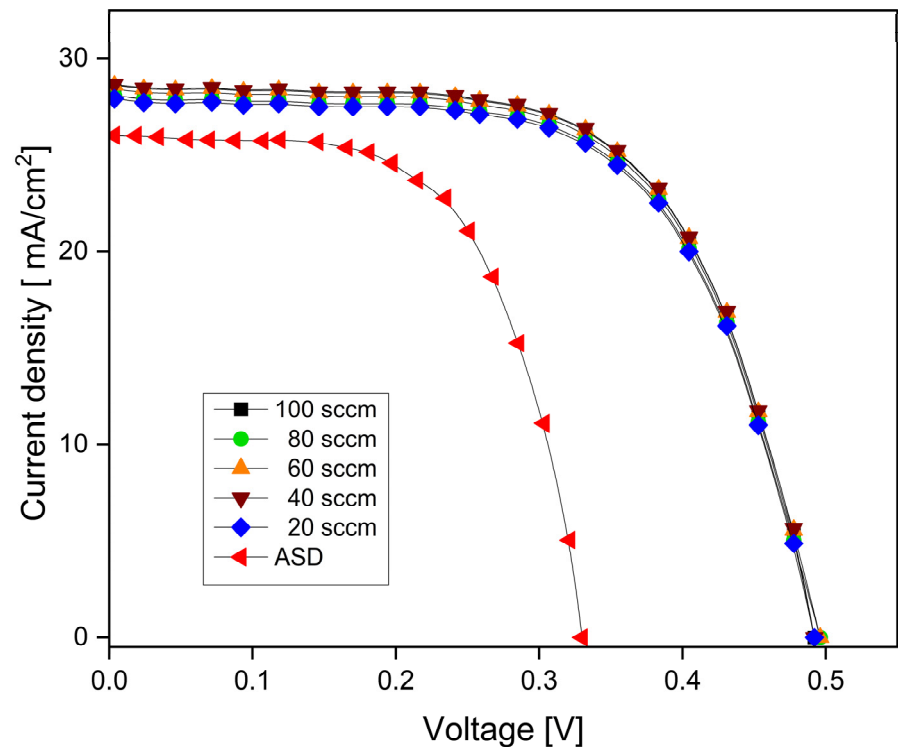


Figure 14. I-V characteristics of dye-sensitized solar cell on ITO-nanorod treated with oxygen plasma of varying gas contents at 20, 40, 60, 80, 100 sccm providing via bias-magnetron RF sputtering process.

Table 1. Photovoltaic parameters of dye-sensitized solar cell based on the ITO-nanorod films treated by oxygen plasma of varying gas contents at 20-100 sccm.

ITO substrate	J _{sc} [mA/cm ²]	V _{oc} [mV]	FF [%]	Efficiency [η, %]
As-deposited	26.8	336	63	6
20 sccm	27.9	491	66	8.6
40 sccm	28.1	493	66	8.7
60 sccm	28.5	494	67	8.9
80 sccm	28.8	497	66	9.2
100 sccm	29.3	500	70	9.5 ¹

¹ The greatest of dye-sensitized solar cell efficient.

Figure 15 shows the overall efficiency of DSSC devices comparing as-deposited film and ITO-nanorod film treated with oxygen plasma in different conditions of gas flow rates. The trend illustrates that when the oxygen gas flow rate is increased, the efficiency of the photovoltaic improves. Compared with as-deposited film, the samples treated with oxygen plasma exhibited higher efficiency, especially at an oxygen gas flow rate of 100 sccm, showing the highest efficiency of 9.5 %. It produces higher energy oxygen plasma with more oxygen gas ions. When falling to ITO-nanorod substrates, the substrate surface is modified by enhancing the roughness of the ITO-nanorod (working electrode) to allow greater dye absorption. This results in better light harvesting efficiency, leading to improved performance of DSSC devices based on ITO-nanorod deposited on a glass substrate.

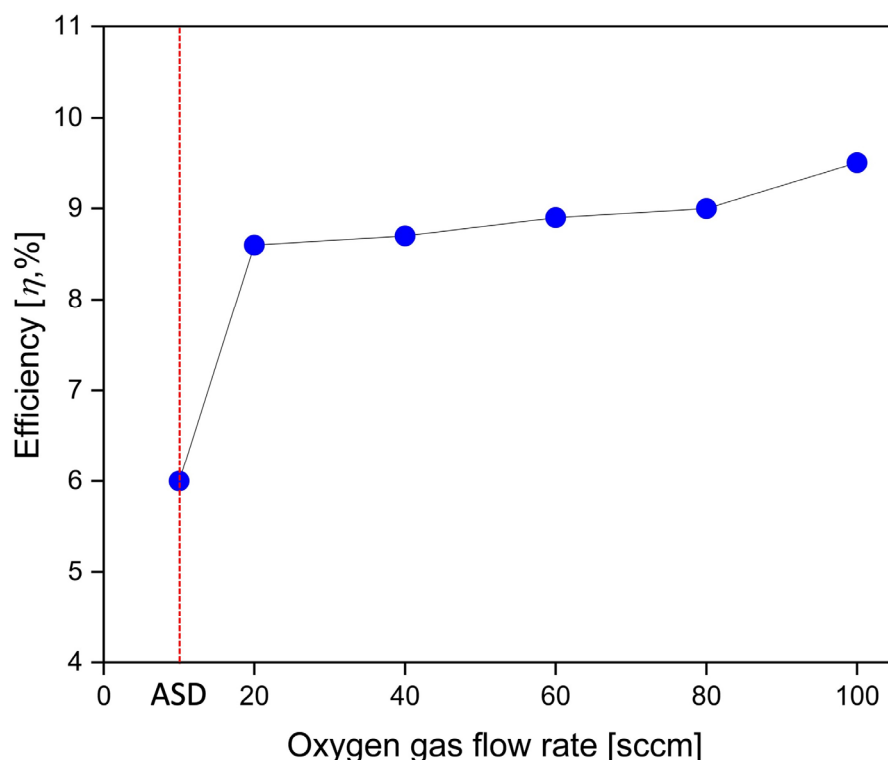


Figure 15. Efficiency of dye-sensitized solar cell of dye-sensitized solar cell on ITO-nanorod treated with oxygen plasma of varying gas contents at 20, 40, 60, 80, 100 sccm providing via bias-magnetron RF sputtering process.

4. Discussion

The study's main objective is to create a new material for working electrodes to achieve DSSC with cost-effective, environmentally sustainable, and simple synthesis. This research emphasizes the critical use of plasma technology to modify the material's surface to investigate a relationship between the structure of the dye and the device's performance. This is necessary to know to develop new devices using environmentally friendly materials. Regarding the proposed stability, the critical issue is to improve the efficiency of solar energy conversion to meet the demand for electricity and energy. Therefore, we propose to improve the efficiency of DSSC based on the use of plasma technology to attack the material surface at the nanoscale level of the working electrode layer. It can be confirmed that plasma technology can penetrate the nanoscale particles to break the bonds and produce suitable properties for application.

The research results found that using plasma to treat the material surface with different gas flow rates affected several properties of the film. At first, in terms of the structure of the film layer, the result shows that when the oxygen gas flow rate is increased, the nanostructure of the ITO layer changes, and the grain size decreases because of the plasma attack on the $\text{In}^{3+}/\text{Sn}^{4+}$ bonds. Secondly, when increasing the oxygen gas flow rate, the result exhibits improved optical properties of the film, which is suitable for use as transparent conductive properties in solar cell devices. Thirdly, when the oxygen gas flow rate increases, the sheet resistance decreases, which is an advantage of the electrical properties that will help the solar cell device transfer charge quickly and reduce energy loss. Finally, when the oxygen gas flow rate increases, the solar cell's efficiency can be increased compared to the average film not treated with plasma.

Therefore, in the near future, we will develop a simple treatment system using low-cost materials such as nitrogen gas or atmospheric pressure to prepare ITO films with better quality and solar cell performance by oxygen plasma treatment.

5. Conclusions

This work successfully fabricated ITO-nanorod films using E-beam evaporation with the glancing angle deposition method. Nanorods illustrated the obtained films. The ITO-nanorod film was treated with oxygen plasma via a bias-magnetron RF sputtering process. To investigate the exceptional result, the oxygen gas flow rate varied by 20, 40, 60, 80, and 100 sccm. Thus, the oxygen plasma treatment has been proven effective in promoting the crystallization of ITO films, resulting in changes in their optical and electrical properties. Notably, the efficiency of DSSC devices can improve through oxygen plasma treatment.

Author Contributions: Conceptualization, W.P. and N.M.; methodology, S.A.; software, T.T.; validation, X.X., Y.Y. and W.P. and N.K.; formal analysis, S.A and T.T.; investigation, A.S.; resources, N.K.; data curation, W.P.; writing—original draft preparation, N.M. and A.S.; writing—review and editing, S.A.; visualization, T.M and K.T.; supervision, N.K. and N.K.; project administration, N.M.; funding acquisition, All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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