Research Article

Structural, Morphological, and Electron Transport Studies of
Annealing Dependent In$_2$O$_3$ Dye-Sensitized Solar Cell

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Indium oxide (In$_2$O$_3$) thin films annealed at various annealing temperatures were prepared by using spin-coating method for dye-sensitized solar cells (DSSCs). The objective of this research is to enhance the photovoltaic conversion efficiency in In$_2$O$_3$ thin films by finding the optimum annealing temperature and also to study the reason for high and low performance in the annealed In$_2$O$_3$ thin films. The structural and morphological characteristics of In$_2$O$_3$ thin films were studied via XRD patterns, atomic force microscopy (AFM), field-emission scanning electron microscopy (FESEM), EDX sampling, and transmission electron microscopy (TEM). The annealing treatment modified the nanostructures of the In$_2$O$_3$ thin films viewed through FESEM images. The In$_2$O$_3$-450°C-based DSSC exhibited better photovoltaic performance than the other annealed thin films of 1.54%. The electron properties were studied by electrochemical impedance spectroscopy (EIS) unit. The In$_2$O$_3$-450°C thin films provide larger diffusion rate, low recombination effect, and longer electron lifetime, thus enhancing the performance of DSSC.

1. Introduction

A commercially viable dye-sensitized solar cell (DSSC) through low-cost processes for electricity generation that exhibits realistic energy-conversion efficiency was first reported by O’Regan and Graetzel in 1991 [1]. In common, DSSCs are composed of a metal oxide semiconductor as a photoanode, organic dyes, electrolyte, and platinum counter electrode [2]. The advantages of DSSCs are low cost, flexibility, being environmental friendly, and fabrication ease that make them be more favorable than conventional silicon solar cells [3]. Furthermore, the performance of the highest reported efficiency (~15%) was achieved by TiO$_2$-based DSSC [4].

However, the evolution of favorable semiconductor materials used as photoanode in DSSCs that show upgraded solar cell performance is still essential. Some photoanodes with higher electron mobility improve the electron transport throughout the semiconductor layer [5]. Many developments have been going on in the recent years in other semiconductor materials for DSSCs, such as ZnO [5], SnO$_2$ [6], and In$_2$O$_3$ [7].

Research on In$_2$O$_3$ is very rare in photovoltaic materials due to its weak photoelectroactivity and poor charge carrier transport in In$_2$O$_3$ [8], thus decreasing the power conversion efficiency of the cell. For example, the previous research on In$_2$O$_3$ as photovoltaic material exhibited a low current density of 0.75 mA/cm$^2$ [9] and 3.83 mA/cm$^2$ [8] which is remarkably low compared to other metal oxides such as TiO$_2$ (9.49 mA/cm$^2$) [10] and ZnO (6.1 mA/cm$^2$) [11]. In spite of that, In$_2$O$_3$ has been used as a dopant in TiO$_2$ to enhance the device performance where it helps to increase the open-circuit voltage ($V_{oc}$) by elevating the energy level of the oxide [12].
2. Experimental Details

2.1. Preparation of In$_2$O$_3$ Thin Film. In$_2$O$_3$ thin films were prepared by using sol-gel method via spin-coating technique as in Figure 1. A stable transparent aqueous solution was formed by adding 0.1M of indium chloride (InCl$_3$) into 50 mL of 2-methoxyethanol. The solution was stirred on a hot plate at 60°C for 24 h. The solution magnetically stirred on a hot plate at 60°C for 24 h. Cooling process for 1 h. Spin-coating technique on FTO glass. Annealed in air at 350°C, 450°C, and 550°C for 30 min. In$_2$O$_3$ thin film.

2.2. Fabrication of DSSC. The dye immersion process is the first step in fabricating the DSSC. Ethanolic N719 dye (0.5 mM) was used to immerse the annealed In$_2$O$_3$ thin films. The thin films were immersed in the dye for 1 day in a glass petri dish. The immersed thin films were rinsed in ethanol to remove excess dye from the FTO substrate. Consequently, the counter electrode (CE) was prepared by depositing platinum paste on a FTO glass substrate by screen printing technique. The platinized FTO is then annealed in air at 400°C for 1 h. The DSSC was fabricated by assembling the annealed CE and immersed photoanode by sandwiching them together. A parafilm layer and two binder clips were used to fix the cell. Finally, the electrolyte (Idolyte MPN 100 Solaronix SA) was injected into the cell. The active area of the resulting DSSC is 1 cm$^2$.

2.3. Characterization of the Thin Films. X-ray diffractometer (XRD) (Siemens D-5000) confirmed the orientation and crystallinity of In$_2$O$_3$ nanostructures. The surface roughness of the thin films was analyzed by atomic force microscopy (AFM) (NTegra Prima). Moreover, the field-emission scanning electron microscope (FESEM) (Zeiss Supra) packed along with energy dispersive X-ray (EDX) unit characterizes morphology and structural properties of the thin films. A transmission electron microscopy (TEM) (Philips, CM21, 20–200 kV) was used to observe the internal structure of the thin films. The photovoltaic performance of the annealed thin films was evaluated through photocurrent density-voltage (J-V) curve measurement via linear sweep voltammetry unit (Gamry Physical Electrochemistry (PHE200)) under 1000 W/m$^2$ illumination (1.5 AM) of OSRAM halogen lamp, 50 W. The electron movement throughout the DSSC was investigated by an electrochemical impedance spectroscopy (EIS) unit (Gamry Series G300 Potentiostat).

3. Results and Discussions

3.1. Structural Characterization. The XRD analysis was carried out with 2θ range of 20° to 60°. Figure 2 shows: The XRD patterns of the annealed In$_2$O$_3$ thin films. The XRD reflections confirm the cubic phase of In$_2$O$_3$ with bravais lattice structure of body-centered cubic, space group of Ia-3, number 206, and Z = 16. The lattice constant of a is 10.117 Å for the bulk In$_2$O$_3$ crystal with cubic structure (JCPDS number 01-071-2194). The diffraction peaks of In$_2$O$_3$ were identified at 2θ = 21.504°, 30.575°, 35.475°, and 51.043°, corresponding to the (h k l) Miller indices of (211), (222), (400), and (440). A preferential orientation along a- was traced at plane (222). The peaks attributing to InCl$_3$ (JCPDS number 00-034-1145) completely disappeared at high annealing temperatures and thus all the diffraction peaks are due to cubic In$_2$O$_3$.
phase. The effect annealing temperature towards the phase transformation from InCl$_3$ to In$_2$O$_3$ can be clearly observed through XRD analysis. The orientation of (111) crystal plane is predominant as the annealing temperature was increased. The crystallinity of the thin films was enhanced as the annealing temperature was increased based on the gradual increase in the intensity of the peaks as observed in the XRD spectrum.

The lattice constant of the cubic structure of In$_2$O$_3$ was calculated using the following equation [13]:

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}},$$

where $a$ is the lattice constant and $d_{hkl}$ is the interplanar spacing. The calculated values of $a$ are listed in Table 1. The values of $a$ decreased and are closer to the bulk one as the annealing temperature was increased from 350°C to 550°C. The large value of $a$ denotes that the unit cell is elongated and compact pressure is in the plane of the film [14]. Yuan et al. mentioned that the increment in annealing temperature caused the compressive strain in the In$_2$O$_3$ film to be relaxed constantly [14]. The film annealed at 550°C is almost fully relaxed as the value of $a$ was smaller than bulk crystal one.

Furthermore, the crystallite sizes of the cubic In$_2$O$_3$ can be calculated using Debye-Scherrer’s formula [15]:

$$D = \frac{k\lambda}{\beta \cos \theta},$$

where $D$ is the crystallite size, $k$ is Scherrer’s constant ($k = 0.94$), $\lambda$ is X-ray wavelength with 0.15406 nm, $\beta$ is the full width at half maximum (FWHM) of the Bragg peak, and $\theta$ is the Bragg angle. The crystallite size of the films increased as the annealing temperature was increased due to enhancement in the crystallinity of the films. The narrow diffraction of the film annealed at 450°C exhibited larger average crystallite sizes than the other annealed films.

Moreover, the number of defects in the thin film can be determined by calculating the dislocation density, $\delta$, by using the following equation [16]:

$$\delta = \frac{1}{D^2}. \quad (3)$$

The dislocation density decreased as the annealing temperature was increased as listed in Table 1. The dislocation density is very large at annealing temperature of 350°C. The annealing treatment with high electron mobility has decreased the amount of defects on the high-annealed film and thus changes the surface structures. The transformation in morphology of the films can be observed through the FESEM images.

### 3.2. Morphological Characterization

AFM analysis shows clearly the morphology characterization to examine the difference in roughness between the thermally treated In$_2$O$_3$ thin films. The previous study reported that AFM is an efficient tool to study the surface morphology and microtopographical properties of thin films [17, 18]. Figures 3(a), 3(b), and 3(c) show the three-dimensional surface morphology of the In$_2$O$_3$ thin films annealed at 350°C, 450°C, and 550°C, respectively.

The RMS values of the annealed thin films were tabulated in Table 2. In$_2$O$_3$ film annealed at 350°C showed around 2 nm of RMS surface roughness with granular structure (Figure 2(a)). The compact nature was seen in the films with high annealing temperature. Low annealed In$_2$O$_3$ thin film was identified as nonhomogeneous structure with a smooth surface area. In contrast, the high annealed films above 350°C displayed rougher structure with RMS value ranging around 11–29 nm (Figure 2(b)). The obtained AFM results were similar to the research of Beena et al. [19]. Hence, AFM analysis denoted that In$_2$O$_3$ thin film annealed at high annealing temperature showed more homogeneous structure with rougher surface roughness compared to low-annealed In$_2$O$_3$ films.

Moreover, the FESEM (Figure 4) results showed roughness structures similar to the results showed in AFM analysis (Figure 3). The In$_2$O$_3$ thin film signifies very apparent smooth surface area or nonhomogeneous structures of In$_2$O$_3$ aggregations in Figure 4(a). In nanoscale (Figure 4(b)) the highly porous In$_2$O$_3$ structure has large nanoparticles in a diameter range of 121 nm. The sphere shape nanoparticles covered with

### Table 1: XRD parameters of the In$_2$O$_3$ thin films annealed at 350°C, 450°C, and 550°C.

| Annealing temperature (°C) | hkl | 2θ (°) | $d_{hkl}$ (nm) | $a$ (nm) | $D$ (nm) | $\delta$ (line$^2$/m$^2$) |
|-----------------------------|-----|--------|----------------|---------|---------|------------------------|
| 350                         | (222) | 30.525 | 0.296 | 10.136 | 8.3810 | $14.237 \times 10^{15}$ |
| 450                         | (222) | 30.550 | 0.292 | 10.129 | 17.188 | $3.385 \times 10^{15}$  |
| 550                         | (222) | 30.625 | 0.292 | 10.105 | 19.101 | $2.741 \times 10^{15}$  |

'InCl$_3$' is X-ray wavelength with 0.15406 nm, $\beta$ is the full width at half maximum (FWHM) of the Bragg peak, and $\theta$ is the Bragg angle.
Table 2: RMS surface roughness of the annealed thin films.

| Annealing temperature (°C) | RMS (nm) | Thickness | Oxygen element (weight %) | Particle size (nm) |
|---------------------------|----------|-----------|---------------------------|-------------------|
|                           |          | FESEM     | TEM                       |                   |
| 350                       | 2         | 346.1 nm  | 19.37                     | 52                |
| 450                       | 11        | 591.7 nm  | 22.37                     | 121               |
| 550                       | 29        | 1.4 μm    | 27.67                     | 410               |

Figure 3: AFM images of In$_2$O$_3$ thin films annealed at (a) 350°C, (b) 450°C, and (c) 550°C.

tiny fur gives a direct pathway to the photogenerated electrons to be diffused easily into the working electrode. Comparatively, the pyramid-like octahedral structure observed in Figure 4(c) due to high-annealing temperature at 550°C shows rougher surface which tallies with the larger RMS value obtained in Table 2. The diameter of the octahedral In$_2$O$_3$ was measured to be 410 nm which is larger than the nanoparticles observed in Figure 4(b). The octahedral structure observed in the FESEM images is proved in the orientation of (111) crystal plane from the XRD spectrum (Figure 2).

The thickness of the film is shown in Figure 4 for thin films annealed at (d) 350°C, (e) 450°C, and (f) 550°C. Thickness of the thin film increased as the annealing temperature increased as seen in Table 2. The EDX images are shown in Figure 5. The presence of chloride is obviously seen in the thin film annealed at 350°C which was also traced in the XRD peaks (Figure 2). The In$_2$O$_3$ thin films annealed at 450°C and 550°C show only indium (In) and oxygen (O) elements from the EDX spectrum. In addition, the EDX spectrum also analyzed the weight % of the oxygen compound which increased gradually as the annealing temperature increases (Table 2).

Table 3: Photovoltaic parameters of In$_2$O$_3$ thin film annealed at 350°C, 450°C, and 550°C.

| Annealing temperature (°C) | $V_{oc}$ (V) | $J_{sc}$ (mA/cm$^2$) | FF | η (%) |
|---------------------------|--------------|----------------------|----|-------|
| 350                       | 0.35         | 2.5                  | 0.32 | 0.28  |
| 450                       | 0.42         | 9.2                  | 0.43 | 1.54  |
| 550                       | 0.36         | 5.8                  | 0.35 | 0.73  |

Furthermore, TEM images in Figure 6 revealed the inner structures of the annealed In$_2$O$_3$ thin films. The particle size measured in the TEM images were more or less similar to the one measured in the FESEM images. From Table 2, the diameter of the In$_2$O$_3$ particles increased as the annealing temperature was increased.

3.3. Photovoltaic Performance of DSSC. Figures 7(a), 7(b), and 7(c) showed the graphical image of the $J$-$V$ characteristics and the corresponding photovoltaic parameters of In$_2$O$_3$ annealed at 350°C, 450°C, and 550°C, respectively. Table 3 lists the corresponding photovoltaic properties. The results
Table 4: Electron transport properties obtained from EIS analysis.

| Temperature (°C) | L (µm) | \( \omega_{\text{max}} \) (Hz) | \( R_\text{ct} \) (Ω) | \( R_L \) (Ω) | \( C_p \) (µF) | \( \tau_{\text{eff}} \) (ms) | \( D_{\text{eff}} \) (cm²s⁻¹) × 10⁻³ | \( L_m \) (µm) | \( \eta \) (%) |
|-----------------|--------|-------------------------------|----------------|-------------|-------------|----------------|----------------|-------------|---------|
| 350             | 0.35   | 1893                          | 26.20          | 215         | 0.16        | 0.53           | 0.00028        | 0.12        | 0.28    |
| 450             | 1.60   | 476                           | 641            | 3.21        | 6.01        | 2.10           | 2.43           | 22.6        | 1.54    |
| 550             | 4.63   | 4755                          | 32.12          | 198         | 0.52        | 0.21           | 0.165          | 1.86        | 0.73    |

Figure 4: FESEM images of In\(_2\)O\(_3\) thin films annealed at (a) 350°C, (b) 450°C, and (c) 550°C and thickness of In\(_2\)O\(_3\) thin films annealed at (d) 350°C, (e) 450°C, and (f) 550°C.

Figure 8 shows the EIS spectra with fitted curves of the annealed thin films. Figure 9 illustrates the magnified version of the film annealed at 450°C. The spectra were fitted based on a transmission line equivalent circuit proposed by early researchers [5, 23–25]. The circuit is shown in Figure 10 and the electron transport properties obtained from the fitted curves are listed in Table 4. The excess thickness in the photoanode layer decreases the performance of DSSCs because of electron recombination that takes place in the cell [22]. Therefore, the optimum annealing temperature for In\(_2\)O\(_3\)-based DSSCs is 450°C with power conversion efficiency (\( \eta \)) of 1.54%.

3.4. Electron Mobility. Figure 8 shows the EIS spectra with fitted curves of the annealed thin films. Figure 9 illustrates the magnified version of the film annealed at 450°C. The spectra were fitted based on a transmission line equivalent circuit proposed by early researchers [5, 23–25]. The circuit is shown in Figure 10 and the electron transport properties obtained from the fitted curves are listed in Table 4. The excess thickness in the photoanode layer decreases the performance of DSSCs because of electron recombination that takes place in the cell [22]. Therefore, the optimum annealing temperature for In\(_2\)O\(_3\)-based DSSCs is 450°C with power conversion efficiency (\( \eta \)) of 1.54%.

Figure 4: FESEM images of In\(_2\)O\(_3\) thin films annealed at (a) 350°C, (b) 450°C, and (c) 550°C and thickness of In\(_2\)O\(_3\) thin films annealed at (d) 350°C, (e) 450°C, and (f) 550°C.

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Figure 5: EDX Sampling of the $\text{In}_2\text{O}_3$ thin films annealed at (a) 350°C, (b) 450°C, and 550°C.

Figure 6: TEM images of $\text{In}_2\text{O}_3$ thin films annealed at (a) 350°C, (b) 450°C, and (c) 550°C.
overall physical structure of the DSSC is illustrated in the model including the capacitances and resistances that exist in the cell [23]. The elements that exist in the cell are sheet resistance \( R_s \), substrate resistance \( R_{FTO} \), substrate capacitance \( C_{FTO} \), transport resistance \( R_t \), charge transfer resistance \( R_{ct} \), chemical capacitance \( C_p \), Warburg impedance \( Z_D \), counter electrode resistance \( R_{Pt} \), and Helmholtz capacitance \( C_{Pt} \) [23–25].

Moreover, the peak frequency of the \( \ln_2 \) semicircles is \( \omega_{\text{max}} \) or \( f_{\text{max}} \) (475.6–4755 Hz). \( \omega_{\text{max}} \) can be presented as follows [26]:

\[
\omega_{\text{max}} = \tau_{\text{eff}}^{-1},
\]

(4)

where \( \tau_{\text{eff}} \) is the electron lifetime of the cell [27]:

\[
\tau_{\text{eff}} = \frac{1}{2\pi f_{\text{max}}},
\]

(5)

In addition, the thickness of the photoanode \( L \) and the electron diffusion length \( L_n \) are related with \( \tau_{\text{eff}} \) and the electron diffusion coefficient \( D_{\text{eff}} \), where [26]

\[
D_{\text{eff}} = \frac{R_{ct}L^2}{R_t\tau_{\text{eff}}},
\]

(6)

\[
L_n = \sqrt{D_{\text{eff}} \times \tau_{\text{eff}}}.
\]

The electron movement across the \( \ln_2 \) layer \( (\omega_{\text{d}}) \) and frequency of the carrier transport process \( (\omega_{\text{rec}}) \) can be described as [24]

\[
\frac{\omega_{\text{d}}}{\omega_{\text{rec}}} = \frac{R_{ct}}{R_t} = \left(\frac{L_n}{L}\right)^2.
\]

(7)

From Table 4, \( \ln_2 \)-based DSSC obtained the longest \( \tau_{\text{eff}} \) of 0.21 ms than the other annealed thin films. The highest \( \tau_{\text{eff}} \) signifies low recombination rate between the excited electrons and the electrolyte layer [28]. In order to have good device performance, the electron diffusion length should be greater than the photoanode length \( (L_n \gg L) \) and the charge transfer resistance should be larger than the transport resistance \( (R_{ct} \gg R_t) \) [29]. Greater \( L_n \) enables the dye molecules to harvest more incident photons [30].

Although the film annealed at high temperature of 550 °C has a better crystalline properties as seen in the XRD spectrum (Figure 2), it showed poor electron transport properties. The same result was observed in Abdullah et al., where the thin films’ crystalline properties increased as the annealing temperature increased but showed shorter electron diffusion length in the respective thin films [10, 31]. It is well known that annealing improves the crystalline properties of thin films by relaxing the compressive strains [14, 32]. However, it does not mean that thin films with greater crystalline properties possess good electron transport. The film annealed at 550 °C obtained shorter \( L_n \) of 1.59 × 10⁻⁶ μm which can be due to greater thickness of photoanode layer \( L \) of 1.4 μm (Figure 4(f)). The excess thickness in the layer can inhibit the DSSC performance by increasing the electron recombination rate in the cell [22].

It was mentioned earlier that, for a desired case in DSSC, the length of excited electron should be the same as or more
than the thickness of the photoanode \((L_n \gg L)\) which is actually to indicate that the electron had reached the working electrode successfully by diffusing through the In\(_2\)O\(_3\) layer with a slow recombination process. In the film annealed at 550°C, it is proven that most of the photogenerated electrons failed to reach the working electrode due to strong recombination process occurring in the cell \((L_n \ll L)\). The strong electron recombination effect in film annealed at 550°C can be observed by analyzing the values of \(R_{ct}\) and \(R_t\). It was mentioned above that \(R_{ct} \gg R_t\) for a good DSSC performance. It is clearly seen that a strong recombination effect takes place in the cell \((R_{ct} \ll R_t)\) and eventually reduces the electron diffusion in the cell and causes small \(D_{eff}\) of 0.473 \(\times\) \(10^{-3}\) cm\(^2\)/s. Bisquert and Fabregat-Santiago outlined \(C_{\mu}\) related with photoanode Fermi level increment \([33]\). The film, annealed at 550°C, obtained 79 \(\mu\)F. Therefore, low \(R_{ct}\) and \(C_{\mu}\) seriously affected the power conversion efficiency of the cell to 0.73%. This type of undesirable case is called Gerischer impedance \([34]\).

In contrast, the In\(_2\)O\(_3\)-450°C caused most of the excited electrons to diffuse through the photoanode layer and reach the working electrode with slow recombination effect. The film obtained larger \(D_{eff}\) around 2.43 \(\times\) \(10^{-3}\) which provides high electron transport rate and thus improves \(J_{sc}\) and \(V_{oc}\) of the cell. Furthermore, the value of \(R_{ct} \gg R_t\) supports the high efficiency in the film annealed at 450°C. The low recombination effect increased more electrons to diffuse into the photoanode. Lower resistance of charge transfer in the cell caused slow recombination of electron-hole and speeds up \(\tau_{eff}\) \([30]\).

Hence, the greater performance of the film annealed at 450°C has faster electron transport with low recombination rate and high diffusion rate. Besides that, annealing plays a major role in enhancing performance of DSSCs where an optimum annealing temperature needs to be known. In this work, the optimum annealing temperature for In\(_2\)O\(_3\)-based DSSC is 450°C. The EIS analysis showed a clear picture of the high and low performance of each annealed thin films.

4. Conclusion

In summary, we have successfully investigated the influence of annealing temperature in In\(_2\)O\(_3\)-based DSSCs. The films showed better crystallinity, rougher structure, and thicker photoanode layer as the annealing temperature increased from 350 to 550°C. The film annealed at 450°C exhibited highest power conversion efficiency of 1.54%. Although, the film annealed at 550°C has better crystalline properties, the greater thickness of the photoanode layer degraded the DSSC performance. Stronger recombination effect was observed in the films annealed at 350°C and 550°C where \(R_t \gg R_{ct}\) with small \(D_{eff}\). The high recombination effect caused slower diffusion of injected electrons in the photoanode. Thus, \(L \gg L_n\). In contrast, the film annealed at 450°C has larger \(D_{eff}\) (2.43 \(\times\) \(10^{-3}\)) and small amount of charge transfer \(R_{ct}\) \(\gg\) \(R_t\), thus increasing \(\tau_{eff}\) (0.21 ms) of the cell. Therefore, the current findings greatly suggested that annealing temperature of 450°C acquires good structural, morphology, and electrical properties for efficient In\(_2\)O\(_3\)-based DSSCs.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.
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