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İsmail Polat

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Thickness effects of Nb2O5 blocking layer on the performance of ZnO-based DSSCs
İsmail Polat, Salih Yılmaz, Tayfur Küçükömeroğlu, Murat Tomakin, Emin Bacaksız
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Thickness effects of Nb_2O_5 blocking layer on the performance of ZnO-based DSSCs

İsmail Polat[1*], Salih Yılmaz[2], Tayfur Küçükömeroğlu[3], Murat Tomakin[4], Emin Bacaksız[5]

[1]Department of Energy Systems Engineering, Faculty of Technology/ Karadeniz Technical University, Trabzon, Turkey
[2]Department of Materials Engineering, Faculty of Engineering and Natural Sciences/ Adana Science and Technology University, Adana, Turkey
[3]Department of Physics, Karadeniz Technical University, Trabzon, Turkey
[4]Department of Physics, Faculty of Arts and Sciences/Recep Tayyip Erdogan University, Rize, Turkey
[5]Department of Physics, Karadeniz Technical University, Trabzon, Turkey
Email: ipolat@ktu.edu.tr[1]; slh_yilmaz@yahoo.com.tr[2]; tayfun@ktu.edu.tr[3]; murattomakin@yahoo.com[4]; eminb@ktu.edu.tr[5]

Abstract

In this paper, Nb_2O_5 thin films with different deposition times of 0, 2, 4 and 6 min were prepared by spray pyrolysis route. Chemical bath deposited ZnO nanorods were grown on Nb_2O_5 thin films. X-ray diffraction pattern indicated that Nb_2O_5 thin films grew in the amorphous phase whereas a hexagonal ZnO structure dominated the pattern. It was also observed that when the production time of Nb_2O_5 thin films was increased, less transparent Nb_2O_5 films were attained. Band gap values of the Nb_2O_5 thin films rised with increment of the production time. To fabricate the DSSCs, FTO/Nb_2O_5/ZnO nanorod hetero-structure were prepared as photo-anodes and counter electrodes were formed by coating Pt on FTO. An electrolyte solution was injected between two electrodes to finish the DSSCs. The best overall efficiency of 0.16 % were reached by a production time of 4 min for Nb_2O_5 thin films serving as blocking layer in ZnO-based DSSCs.

Keywords: Nb_2O_5 thin film, ZnO nanorods, blocking layer, DSSCs.

1. INTRODUCTION

Dye-sensitized solar cells (DSSCs) which have many advantages such as simple structure, low cost manufacturing and fabrication on flexible substrates have been considered as potential alternative to Si-based solar cells [1]. To improve the performance of DSSCs, different materials such as TiO_2 [2], ZnO [3], SnO_2 [4], and Nb_2O_5 [5] have been used by many research groups, and its efficiency has reached to 12.3% by using nanocrystalline TiO_2 [6]. In these materials, ZnO has attracted more attention as an alternative to TiO_2 for DSSCs due to its electron affinity, higher electronic mobility, and large exciton binding energy (60 meV) [7-9].

DSSCs typically consist of four interface including transparent conduction oxide/photoanode material, photoanode material /photosensitizer, photosensitizer/electrolyte, and electrolyte/counter electrode. During the operation of DSSCs, the electrons need to pass through these interfaces. However, some of the photo-injected electrons may recombine at the electrolyte/electrode interface because of absence of energy barrier layer [10]. The charge recombination brings about a decrease in the photo-current and hence efficiency of DSSC is negatively affected [1,12]. The charge recombination rate can be significantly minimized by introducing blocking layer between transparent conduction oxide andphotoanode materials [13-14]. A number of photo-anode materials such as TiO_2, Nb_2O_5, and ZnO, etc. can be used as a blocking layer in the fabrication of DSSCs [15-16]. Among these materials, Nb_2O_5 which has an n-type semiconductor with a band gap of 3.4 eV is remarkable due to its physical properties such as excellent chemical stability, very low optical absorption in the IR and NIR region, corrosion resistance, and high refractive index.

In this report, we investigated thickness effects of blocking layer on the photovoltaic properties of ZnO-based DSSCs. Nb_2O_5 thin film was used as a blocking layer and produced by the spray pyrolysis method. Its thickness was controlled by deposition time. ZnO nanorods grown by CBD technique was used as photo-anodes. Grown samples were characterized by XRD and photo-spectrometer. Photovoltaic properties of the fabricated DSSCs were determined using current density and voltage (J-V) curves under solar simulator.

(*) Corresponding author

2. EXPERIMENTAL DETAILS

2.1 Grown process of Nb_2O_5 blocking layer and ZnO nanorods

Nb_2O_5 blocking layer was deposited on glass substrates and fluorine tin oxide (FTO) coated glass substrates by using spray pyrolysis method at different deposition time of 0, 2, 4, 6 min. Prior to deposition, substrates were ultrasonically cleaned with soap, ethanol and distilled water for 10 min, respectively. To obtain Nb_2O_5 blocking layer, niobium (V) chloride solution with a concentration of 0.02 M was prepared in a mixture of de-ionized water (89 mL), hydrogen peroxide (10 mL) and acetic aside (1 mL) and then prepared solution was sprayed on both glass and FTO substrates at 400 °C. During the growth, the plate of substrates was rotated with 10 rev/min to obtain homogeneous film surface. After deposition, Nb_2O_5 thin films were annealed at 500 °C for 60 min in air and then ZnO nanorods were grown on FTO substrates and Nb_2O_5 covered FTO substrates by the CBD technique. The details of CBD technique for the synthesis of ZnO nonorods can be found in our previous work [17]. Briefly, total deposition time and solution temperature to grow ZnO samples were adjusted as 60 min and about 80-85 °C, respectively.

2.2 Fabrication of DSSCs

ZnO nanorods prepared on both FTO substrates and Nb_2O_5 covered FTO substrates, whichwere used as working electrodes were submerged in a 0.5 mM of a solution consisting of absolute ethanol and N719 dye remained under the dark condition for 24 h at room temperature (RT). The obtained working electrodes were rinsed with ethanol and dried at RT. To form counter electrode, Pt solution was covered by spin coating technique on FTO substrates and then counter electrodes were annealed at 450 °C for 10 min. A spacer with 60 µm thick was placed between two electrodes in order to seal the electrolyte solution. The active area of each electrode was adjusted as 0.20 cm^2. After preparation of two electrodes, a hot-pressing technique at 95 °C was applied to two elecrodes to come together and then a few drops of Iodolyte AN-50 electrolyte solution was injected to complete the fabrication of DSSCs (Figure 1).

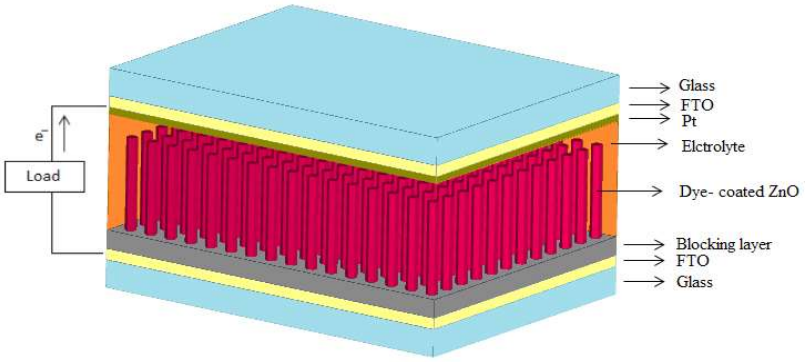

Figure 1: Schematic view of the fabricated ZnO-based DSSCs

2.3 Characterization

X-ray diffraction (XRD) data was obtained using Rigaku D/Max-IIIC diffractometer with CuK radiation over the range of 2θ = 20-60° at RT. SpectraMax M5 was used to investigate optical properties of the samples in the wavelength range of 190-1100 nm. Electrical measurements were carried out using a Keithley 2401 source-meter under illumination of 100 mW/cm^2.

3. RESULTS AND DISCUSSION

In order to investigate the structural features of the obtained specimens, XRD measurements were taken. XRD pattern of unannealed and annealed Nb_2O_5 thin film on glass substrate for deposition time of 6 min is shown in Fig. 1(a) and (b), respectively. As can be seen from the Figure 2, both unannealed and annealed films don't have any diffraction peaks. It was understood that thickness of the Nb_2O_5 thin film with deposition time of 6 min was not enough to start crystal structure, meaning that unannealed and annealed Nb_2O_5 films grew in the amorphous phase. Coşkun and Demirel synthesized Nb_2O_5 thin films by using RF magnetron sputtering system on glass substrates under pressures of 3.0, 6.0, 12.0 and 15.0 mTorr and they obtained that the samples didn't indicate any diffraction peak in the XRD results [18]

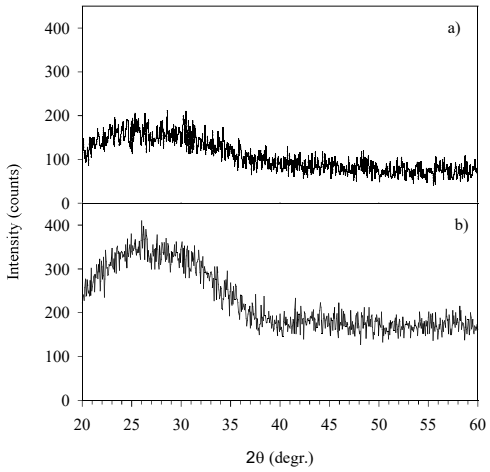

Figure 2: X-ray diffraction patterns of Nb_2O_5 thin film on glass substrates for deposition time of 6 min: (a) unannealed, (b) annealed at 500 °C for 60 min in air.

Figure 3(a-d) displays XRD pattern of FTO/Nb_2O_5/ZnO structure. This spectrum was obtained after Nb_2O_5 thin film was grown by spray pyrolysis method for different deposition times of 0, 2, 4 and 6 min on FTO substrates and then ZnO nanorods was deposited by CBD technique on FTO/Nb_2O_5 structure. XRD pattern of FTO/ZnO without Nb_2O_5 layer is given in Figure 3a. It is seen from Figure 3a that peaks indexed to (100), (002), (101) and (102) belong to wurtzite structure of ZnO (JCPDS card no: 36-1451) with a preferred orientation of (002) plane. It is also observed that the peaks labeled as # relate to FTO substrate (JCPDS card no: 00-041-1445). Figure 3(b-d) shows the XRD pattern of FTO/Nb_2O_5/ZnO structure with Nb_2O_5 deposition times of 2, 4 and 6 min, respectively. It can be noticed from these figures that hexagonal structure of ZnO was conserved during all the patterns. With increasing deposition time of Nb_2O_5 thin films from 2 min to 6 min in the FTO/Nb_2O_5/ZnO structure, there were no significant change in peak position and orientation of ZnO. In addition to this, any peaks of metallic Nb or Nb compounds were not observed in the pattern within the detection limit of XRD.

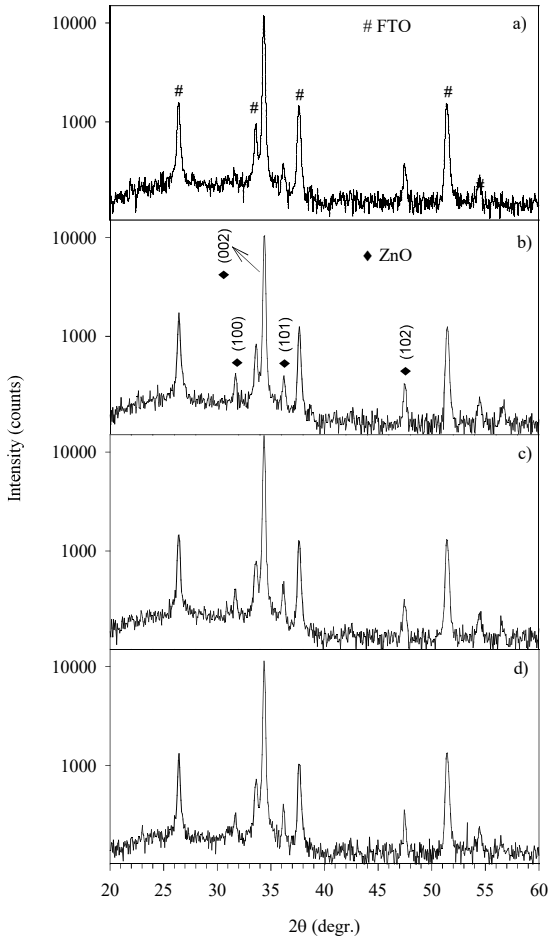

Figure 3: X-ray diffraction patterns of FTO/Nb_2O_5/ZnO structure with Nb_2O_5 deposition time of (a) 0 min, (b) 2 min, (c) 4 min, and (6) min.

In order to investigate optical properties of blocking layers, transmittance spectra of Nb_2O_5 thin films at deposition times of 2, 4 and 6 min on glass substrates were recorded in the range of 300-1100 nm at RT and the resultant data were given in Figure 4. It is clearly noticeable from Figure 4 that transmittance was strongly affected by deposition time in the range of 550-900 nm. The average transmittance decreased about from 70 % to 35 % in the same range, once the deposition time of Nb_2O_5 thin film increased from 2 min to 6 min. The decrement in the transmittance of Nb_2O_5 thin film could be attributed to the increment thickness of Nb_2O_5 thin films and scattering of light at the grain boundaries of Nb_2O_5 [19].

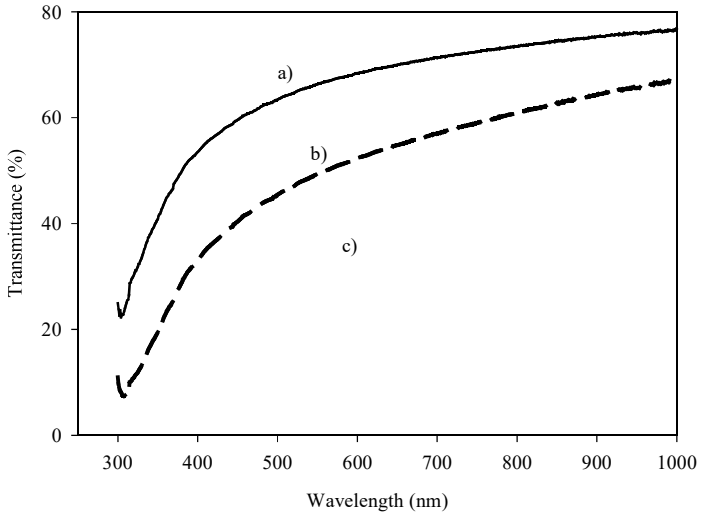

Figure 4: Transmittance spectra of Nb_2O_5 thin films for deposition time of (a) 2 min, (b) 4 min and (c) 6 min.

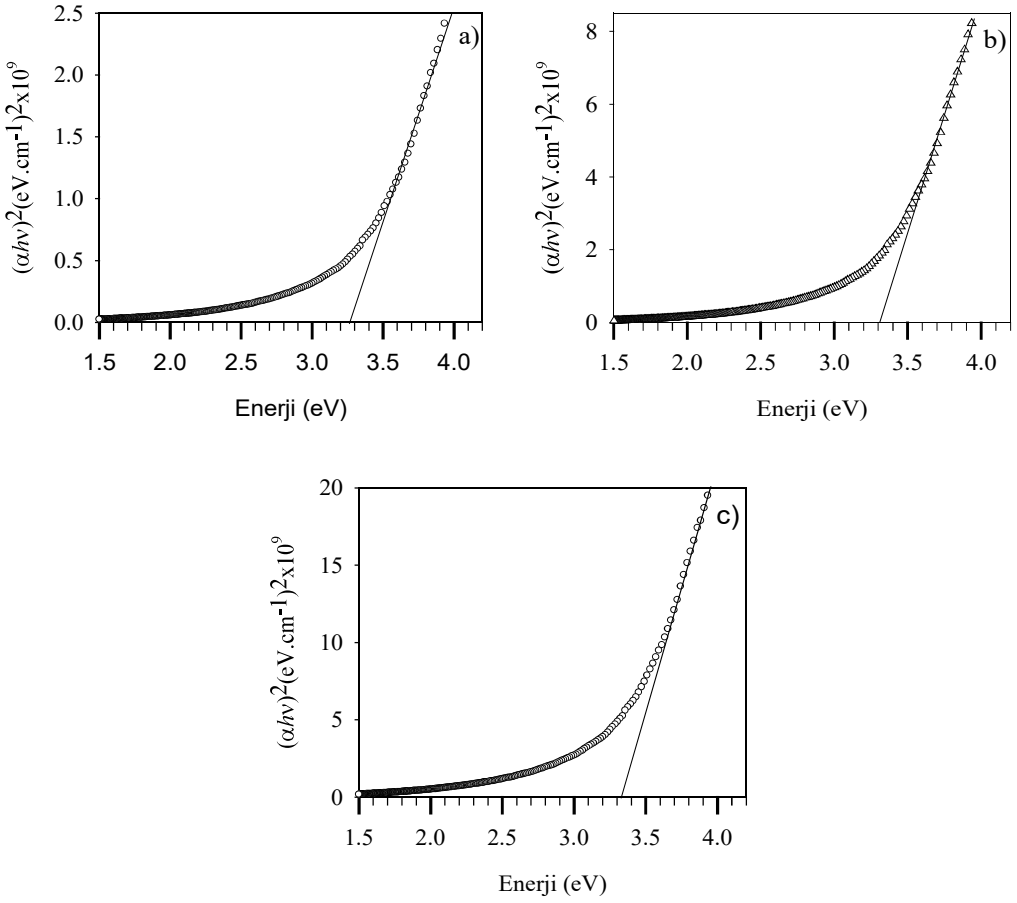

Figure 5: Tauc plots of Nb_2O_5 thin films on glass substrates for deposition times of (a) 2 min, (b) 4 min, and (c) 6 min.

In order to obtain information about the band gap energy (E_g) of Nb_2O_5 thin films prepared on glass substrates for different deposition times, Tauc's plot analysis was applied and results were shown in Figure 5. It is seen from Figure 5 that E_g values of Nb_2O_5 thin films with the deposition times of 2 and 4 min were found to be almost 3.25 eV and 3.30 eV,

respectively. With increasing deposition time up to 6 min, E_g value slightly increased to 3.32 eV, approaching the bulk value of Nb_2O_5 crystal (3.40 eV) [20]. This can be explained by the increment thickness of thin film with increasing deposition time. The band gap energy can be changed through various factors such as deposition technique, doping with foreign atoms, lattice strain, grain size, carrier concentration, and stoichiometric ratio. Dash et al. prepared Nb_2O_5 thin films using sputtering system and they determined the band gap energy to be 3.65 eV [21]. Liu et al. produced Nb_2O_5 nanorods and Nb_2O_5 nanotubes and they found E_g value to be 3.97 and 3.72 eV, respectively [22].

To investigate the photovoltaic performance of DSSCs, the current density-voltage (J-V) curves were taken under sun simulator with AM 1.5 G filter. Figure 6 indicates J-V curves of the fabricated DSSCs and their short circuit current density (J_{SC}), open circuit voltage (V_{OC}), fill factor (FF), and efficiency (η) are listed in Table 1. It is found that DSSC without blocking layer has a J_{sc} of 0.67 mA/cm^2, V_{oc} of 0.55 V, corresponding to η of 0.11 %. With inserting of Nb_2O_5 thin film between FTO and ZnO nanorod layers as a blocking layer for deposition time of 2 min, though there seems no variation in the V_{oc} value of DSSC, J_{sc} value enhances to 0.73 mA/cm^2. On the other hand, the production time of Nb_2O_5 thin film reaches to 4 min, J_{sc} and V_{oc} values improves up to 0.76 mA/cm^2 and 0.56 V, respectively and its efficiency increases to 0.16 %. The reason for the increment in efficiency may be explained by the potential barrier at the interface of FTO and ZnO nanorod layers. This potential suppresses electron leakage between two layers and enhances the current density [23]. Lim et al. investigated the influence of a Nb_2O_5 blocking layer on TiO_2 based DSSCs. They stated that the efficiency of fabricated DSSC increased with the increase of deposition time of blocking layer [16]. However, further increase in deposition time of 6 min for blocking layer cause a reduction in efficiency of DSSC (η=0.10 %). The reason for reduction is related to small value of J_{sc}. An excessively thick blocking layer with increasing deposition time decreases dramatically transfer rate of photo-generated electrons [24]. It is known that thick blocking layer deteriorates the transmittance of Nb_2O_5 thin film in the visible region, which is consistent with our transmittance results.

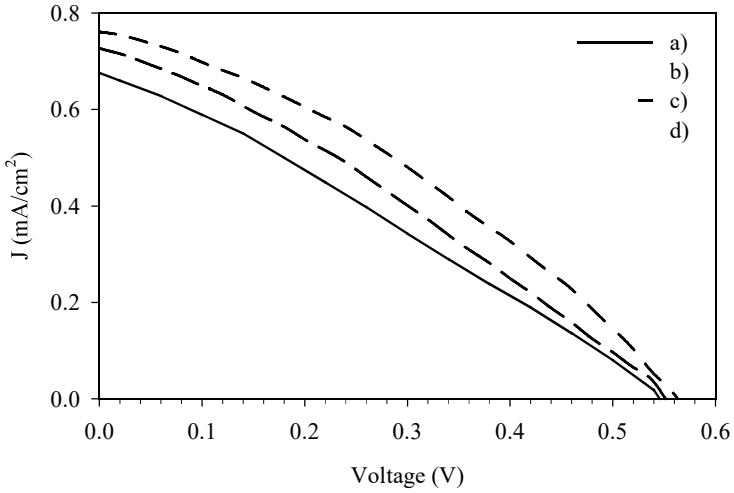

Figure 6: The J-V curves of ZnO-based DSSCs (a) without blocking layer, with blocking layer deposited at 2 min (b), 4 min (c), and 6 min (d).

Table 1: Performance of fabricated DSSCs with and without blocking layer deposited at 2 min 4 min and 6 min.

Deposition time of Nb_2O_5 thin film (min)	J_{sc} (mA.cm^{-2})	V_{oc} (V)	FF	η (%)
0	0.67	0.55	0.29	0.11
2	0.73	0.55	0.30	0.13
4	0.76	0.56	0.34	0.16
6	0.62	0.58	0.28	0.10

4. CONCLUSIONS

Nb_2O_5 thin film was produced by spray pyrolysis method for different deposition times, and used as a blocking layer in DSSCs. ZnO nanorods was grown by CBD technique on FTO substrates and Nb_2O_5 coated FTO substrates and used as a photo-anodes. From the XRD results, both unannealed and annealed Nb_2O_5 thin films grown on glass substrates for deposition time of 6 min have amorphous structure. XRD pattern of FTO/ZnO and FTO/Nb_2O_5/ZnO structure revealed that ZnO nanorods had a hexagonal structure with a preferred orientation of (002) plane. With introduction of blocking layer between FTO andZnO, there were no significant change in peak position and orientation of ZnO. Moreover, any peaks of metallic Nb or Nb compounds were not observed in the pattern. From optical measurements, the average transmittance decreased consistently almost from 70 % to 35 % in the range of 550-900 nm, as deposition time of Nb_2O_5 thin film increased from 2 min to 6 min. E_g values of Nb_2O_5 thin films determined to be 3.25 eV and 3.32

eV for the deposition time of 2 min and 6 min, respectively. From the electrical measurements, maximum conversion efficiency was found to be 0.16 % with J_{sc} of 0.76 mA/cm^2, V_{oc} of 0.56 V, and FF of 0.34 for deposition time of 4 min in DSSC.

6. ACKNOWLEDGEMENTS

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Preparation and characterization of CdS:Ga and CdS:(Ga, K) thin films via spray pyrolysis

Saltuk Buğra Töreli[1*], Salih Yılmaz[2], İsmail Polat[3], Murat Tomakin[4], Emin Bacaksız[5]

[1] Adana Science and Technology University, Graduate School of Natural and Applied Sciences, Nanotechnology and Engineering Sciences, Turkey
[2]Adana Science and Technology University, Faculty of Engineering and Natural Sciences, Department of Materials Engineering, Turkey
[3]Karadeniz Technical University, Faculty of Technology, Department of Energy Systems Engineering, Trabzon, Turkey
[4]Recep Tayyip Erdoğan University, Faculty of Arts and Sciences, Department of Physics, Rize, Turkey
[5]Karadeniz Technical University, Faculty of Sciences, Department of Physics, Trabzon, Turkey
Email: saltuktoreli@gmail.com

Abstract

Cadmium sulfide (CdS) is an II-VI group compound semiconductor and it has a widespread usage in many application areas in the industry. The objective of this work is to reveal CdS samples which exhibit the best optical and electrical behavior when samples were doped as single (Ga) and dual (Ga and K) elements at various levels. Spray pyrolysis route was used to synthesize $Cd_{1-x}Ga_xS$ and $Cd_{1-x-y}Ga_xK_yS$ thin films. All thin films were characterized in terms of structural, morphological, chemical, optical and electrical properties by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), transmittance, photoluminescence (PL), Hall effect and resistivity measurements. Single doping to CdS with Ga was the first step of the study and Ga concentrations were applied as 2, 4, 6 and 8 at.%. It was determined that the highest carrier concentration was displayed by 2 at.% Ga-doped CdS sample as 9.91 x 10^{12} cm^{-3}. Transmittance value of this sample was also found above 60% after 540 nm. Therefore, it was decided that 2 at.% Ga-doping was to be constant content for the second step of the study (x=0.02). As for dual doping, K level was selected as 1, 2, 3, 4 and 5 at.%. Among all (Ga-K)-co-doped CdS thin films, 4 at.% K-doped sample exhibited the best optical and electrical properties when considering the following results; the carrier concentration was as high as 5.90 x 10^{12} cm^{-3} and optical transmittance was the highest one, at least 80% in the range of 540-800 nm. It was also noted that K-dopings mostly brought about an enhancement in the transmission. In conclusion, it was obtained that 4 at.% K-doped $Cd_{0.98}Ga_{0.02}S$ thin films could be a good candidate for the solar cell applications.

Keywords: CdS, Ga-doped CdS, (Ga-K)-co-doped CdS, spray pyrolysis.

(*) Corresponding author