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DC-Magnetron Sputtered Mo Back Contact for Chalcopyrite Thin Film Solar Cells

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In present work, Mo films were deposited on corning glass substrates using DC-Magnetron sputtering. Influence of DC sputtering power on electrical, structural, morphological, optical and topological properties has been investigated by using Hall effect, X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), UV-Visible spectroscopy, non-contact-atomic force microscopy (NC-AFM) etc. It is observed that electrical resistivity and adhesion of Mo thin film were strongly affected by DC power. The synthesized Mo films were of few micrometer thicknesses (~ 0.9-1.6 μm) with deposition rate in the range of 32-57 nm/min. Cross-hatch cut and Scotch tape adhesion test showed that all Mo films have good adhesion to the substrate. XRD analysis showed that as-deposited Mo films have preferred orientation in (110) direction and with enhancement in its crystallinity and average grain size with an increase in the DC sputtering power. Furthermore, XRD analysis showed that the Mo films deposited at DC sputtering power < 300 W exhibit tensile strain, while deposited at DC sputtering power > 350 W – exhibit compressive strain. FE-SEM analysis showed that all Mo films are dense, homogeneous and free of flaws and cracks. In the visible range of the spectrum, an increase in an average reflection of Mo films with DC sputtering power was observed by UV-Visible spectroscopy analysis. NC-AFM characterization revealed that the surface roughness of the films increases with an increase in the DC sputtering power. Hall effect measurements showed that the electrical resistivity of Mo films decreases while charge carrier mobility show increasing trend with increase in DC sputtering power. The obtained results suggest that as-synthesized Mo thin films with DC power < 300 W have potential application as a back contact material for chalcopyrite compounds based on solar cells due to good adhesion and low electrical resistivity.

Keywords: Molybdenum, Resistivity, DC sputtering, AFM, XRD.

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1. INTRODUCTION

As on today, silicon (Si) based technology has the lion's share in solar cell production. However, Si solar cells and semiconductor technology requires the use of clean room and high thermal treatment during the fabrication. Several efforts have been made to reduce the expensive raw material consumption in cell production and to enhance the efficiency of the solar cells. However, the use of expensive raw material and clean process along with high temperature thermal treatment makes it high production cost which leads to the limitation in application of Si solar cells. Now a day, thin film photovoltaic's become extremely essential technology due to use of inexpensive raw material and no requirement of high temperature thermal treatment. Among the various solar cells technologies, dye-sensitized solar cell (DSSC)[1] and chalcopyrite compounds based technologies are considered as the alternative choice for the conventional Si technology due to the advantages of low cost and ease of fabrication from stable and earth's abundant materials. In comparison with Si cells, the DSSC has the ability to absorb more radiation per area. The flexible cells can be realized for various applications that are not applicable in rigid Si cells. The chalcopyrite compounds solar cells such as CIS, and CIGS are the most promising materials as absorbing layers for thin-film solar cells due to their high optical absorption of solar spectrum, feasibility of band gap engineering, low material cost, excellent long term stability, high radiation resistance, large-area production and great potential for developing high-efficiency flexible solar cells [2, 3]. These solar cells have achieved remarkable high power conversion efficiencies ~ 20 % [4] In spite of the high efficiency, the elements used such as In and Ga are rare and expensive. Recently, a novel material CZTS has been explored in place of CIGS in research field. The CZTS is the promising candidate material because of low cost, earth abundant, non-toxic elements and optimal band gap ~ 1.4-1.5 eV with high absorption coefficient 10^4 cm^{-1} [5].

In DSSC and CZTS thin film solar cells, metal back contact plays crucial role to collect charge carriers. It collects the charge carriers from the absorber layer and transports them to the external load [6]. The prerequisite properties for the metallic materials as the back contacts include [7]; i) Good adhesion to the substrate, ii) High conductivity or low resistivity, iii) Must form an Ohmic contact with the CIGS absorbers or back of a DSSC, iv) Thermal expansion coefficients should matched with the substrate, v) Should serve as a proper transport for charge carriers, vi) Must be chemical inert and, vii) Should be stable during the deposition processes. Many research groups have studied the metal back contact for solar cells. These include Molybdenum (Mo), Platinum (Pt), Gold (Au),Copper (Cu), Silver (Ag) [8], Tungsten (W), Tantalum (Ta), Niobium (Nb), Vanadium (V), Cromium (Cr), Titanium (Ti), Magnesium (Mn)[9], etc. Each metal back contact

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layer showed advantages and limitations when used in CZTS solar cells. For example Orgassa et al. [9] showed that metals such as Ti, V, Cr and Mn might react with Se during the deposition of CIGS films. Metals like Au as well as Pt show significant diffusion into the CIS films when annealed at elevated temperatures [10]. In addition, some metals such as Pt, Au and Mo could form moderately low resistance contacts with CIS films [10]. It has been reported that only Mo can form suitable ohmic contacts for chalcopyrite compounds based solar cells [11] due to its encouraging properties such as chemical inertness, high conductivity, low recombination rate for minority carriers, barrier layer to impair the diffusion of impurities into the absorption layer, relative stability at the processing temperature and resistance to alloying with Cu, Zn and Sn [8]. Moreover, Mo allows the ohmic contact with CZTS/Se which leads to the formation of an interfacial layer $MoSe_2$ in between absorber layer and Mo back contact thereby improving the solar cell performance [12]. The formation of the $MoSe_2$ layer depends not only upon selenization conditions but also on the deposition parameters of the Mo thin films [13, 14].

There are some reports which indicated that Mo films prepared at high working gas pressures are under tensile stress, they have high resistivity with good adhesion, while those prepared at low working gas pressure are under compressive stress and have low resistivity but poor adhesion [11]. Martinez and Guillen [11] reported that RF-sputtered Mo films have comparable electrical properties and to obtain densely packed structure and to have minimum stresses, it is necessary to have low RF-power densities. Gordillo et al. [8] reported that low argon partial pressure and high DC sputtering power is necessary to obtain low resistivity Mo films. These experimental results showed that the preparative techniques and the processing conditions have a strong influence on the microstructures and physical properties of the Mo thin films. With this motivation an attempt has been made in the present study to investigate the influence of DC sputtering power on the adhesion, structural, optical, morphology and electrical of Mo thin films. We found that these properties critically depend on applied DC sputtering power.

2. EXPERIMENTAL

2.1 Film Preparation

Molybdenum (Mo) thin films were deposited on cleaned corning # 7059) substrate (2 cm×5 cm) by using commercial planar DC magnetron sputtering unit (Model: 12" MSPT, Hind High Vacuum) using a target of 3 inch diameter (99.99 %, Vin Karola Instrument, USA). Substrates were initially cleaned using soap and piranha solution and then by double distilled water with ultrasonic bath for 30 min. The substrates were dried by blowing with hot air. Immediately after drying, the substrates were introduced into the vacuum chamber and subsequently deposition was performed at various DC powers. Prior to deposition, the vacuum chamber was evacuated to a base pressure of $2{\cdot}10^{-5}$ mbar using rotary and diffusion pump. The target-to-substrate distance was maintained at 90 mm and the deposition was carried out for 30 min. All Mo films were grown at a constant substrate temperature of 400 °C with a water cooled target. During the deposition, pure argon (99.99 %) was introduced in vacuum chamber to maintain a pressure. A series of Mo thin films were sputtered under different DC powers from 200 W to 350 W by keeping all other parameters constant. All deposition parameters are listed in the Table 1.

2.2 Film Characterization

The structure and crystallinity of Mo thin films were investigated by using a X-ray diffractometer (Bruker D8 Advance, Germany) equipped with CuK_α line ($\lambda = 1.54$ Å). The surface morphology of Mo thin films was analyzed by using field emission scanning electron microscope (FE-SEM) (Hitachi, S-4800, Japan) with the operating voltage of 15 kV. The thickness of Mo thin films was estimated by using cross sectional SEM (JEOL JSM-6360A) with operating voltage of 20 kV. The surface topographical investigation was carried out by using non-contact atomic force microscopy (NC-AFM) (JEOL, JSPM-5200). The reflection spectra were measured by using UV-Visible spectrophotometer (JASCO, V-670) in the range 400-800 nm. The electrical resistivity of Mo films was calculated by using Ecopia Hall Effect measurement (HMS-3000) set up. Thickness of films was determined by surface profiler (KLA Tencor, P-16+) and also confirmed by FE-SEM analysis.

Table 1 – Deposition parameters used to synthesize Mo thin films by using DC-Magnetron sputtering

Process Parameter	Value	Unit
Working gas pressure	0.2	mbar
Target-to-substrate distance	9	cm
Deposition power	200-350	Watt
Substrate temperature	400	°C
Base pressure	$2{\cdot}10^{-5}$	mbar
Deposition time	30	min

3. RESULTS AND DISCUSSION

3.1 Adhesion Test of Mo Thin Films

The adhesion test property of Mo thin films to the corning glass substrate was investigated by Cross-hatch cut and Scotch tape test. A slight decrease in adhesion of Mo thin films to the corning glass substrate has been observed when DC sputtering power increased from 200 W to 350 W. The degradation of the adhesion property of Mo thin films with increasing DC sputtering power can be attributed to increase in deposition rate of Mo on the substrate (discussed later). Due to high deposition rate the ad-atom does not get enough time to strongly adhere to the substrate. These results are consistent with previously reported results [15]. Wang et al. [16] and Yoon et al. [17] reported the effect of the residual stress on the adhesion of the thin film. They reported the relationship between the residual stress and the working pressure for the sputtered thin films. The residual stress changes from the compressive stress to the tensile stress with the increasing working

pressure. The excessive compressive or the tensile stress can result in the poor adhesion between the Mo film and the substrate.

3.2 Variation of Deposition Rate

Films were deposited for a desired time period (30 min) and deposition rate is then calculated from thickness measurement. The variation of deposition rate as a function of DC sputtering power is shown in Fig. 1. As seen from the figure, the deposition rate linearly increases with increasing DC sputtering power. It increases from 32 nm/min to 57 nm/min when DC sputtering power increased from 200 W to 350 W. The inset table shows thickness of Mo films and applied DC sputtering power.

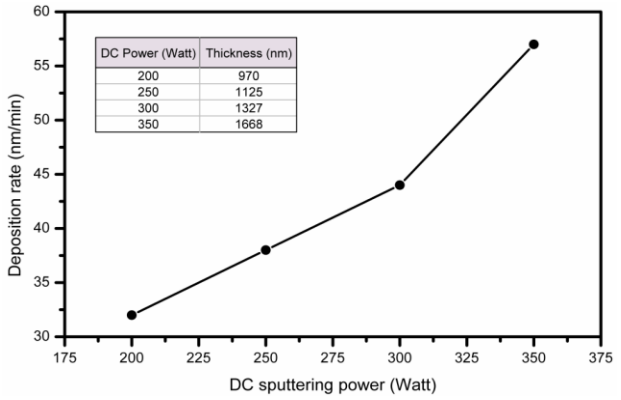

Fig. 1 – Variation of deposition rate of Mo films as a function of DC sputtering power. The inset table shows the thickness of Mo thin films and applied DC sputtering power

The increase in deposition rate can be attributed to increase in density of sputtered Mo atoms with increase in DC sputtering power. With increase in DC sputtering power the kinetic energy of Ar+ ions impinging on Mo target increases which results ejection of large number of Mo atoms from the target. Consequently, the deposition rate increases with increase in DC sputtering power. Increase in deposition rate with increase in DC sputtering power for Mo thin films deposited by DC-Magnetron sputtering has been reported previously by Khan and Islam [18].

3.3 X-ray Diffraction Analysis

The structural investigation such as crystal structure, preferred orientation of crystal, grain size and strain of Mo thin films were investigated from x-ray diffraction (XRD) analysis. Fig. 2 shows X-ray diffraction pattern of Mo thin films deposited on glass substrate at different DC sputtering power. The structural investigation such as crystal structure, preferential orientation of crystal, grain size and strain of Mo thin films were investigated from XRD analysis. All XRD patterns were obtained at a grazing angle of 10 and at step size of 0.5 s. The crystallites of Mo thin films have the cubic crystal structure (JCPDS card # 00-042-1120) with preferential orientation along (110) direction. At higher deposition power 350 W, (200) and (211) planes was observed with very small intensity compared to (110) diffraction plane. With increase in DC sputtering power the line-width (Full width at half maximum, FWHM) of diffraction plane (110) decreases and, its sharpness and intensity increases implying increase in its average grain size ($d_{x\text{-}ray}$) and the crystallanity. The grain size was estimated by using Debye-Scherer's formula [19],

$$d_{x\text{-}ray} = \frac{0.9\ \lambda}{\beta \cos\theta_B}, \qquad (1)$$

where λ is the wavelength of diffracted radiation, θ_B is the Bragg angle and β is FWHM in radians.

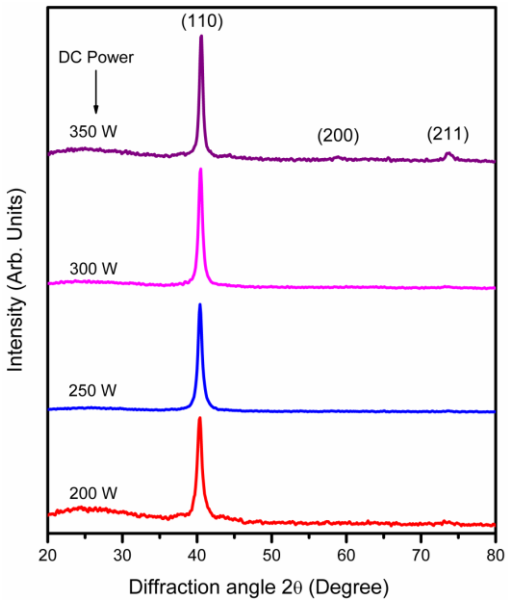

Fig. 2 – Low angle XRD patterns of Mo films deposited at different DC sputtering powers

It is observed that with increase in DC sputtering power the average grain size increases (see Table 2). Furthermore, with increase in DC sputtering power the diffraction peak (110) slightly shifts towards higher diffraction angle. The shifting of peak to higher diffraction angle can be attributed to strain in the films [20]. The intrinsic strain (%) in the Mo film was calculated from the (110) diffraction peak by using following equation [14]:

$$Strain\ (\%) = \frac{a - a_0}{a_0} \cdot 100, \qquad (2)$$

where a_0 and a represent the reference lattice constant (0.3147 nm, JCPDS card # 42-1120) and lattice constant of the Mo films obtained from the XRD measurements, respectively. The estimated values of the lattice constants and the strain in the DC-Magnetron sputtered Mo films are listed in Table 2.

As seen from Table 2, the Mo films deposited at DC sputtering power > 300 W exhibit tensile strain and its value decreases with increase in DC sputtering power, while the Mo film deposited at DC sputtering power of 350 W and more exhibit compressive strain. The origin of the strain in the sputtered Mo films may be related to several factors, including voids, oxygen or argon im-

Table 2 – XRD analysis of Mo films deposited using DC-magnetron sputtering

DC Power, W	Grain size, nm	Lattice constant, nm	Strain, %
200	11.1	0.3155	0.25
250	13.0	0.3152	0.15
300	14.1	0.3149	0.06
350	14.3	0.3143	– 0.12

purities, and crystallographic flaws [16]. The high strain on Mo films may cause adhesion problems and compromise its long term reliability. It is worth mentioning here that Mo films deposited at high DC power (400 W) shows poor adhesion and they can be easily wiped out even by rubbing tissue paper on the substrate.

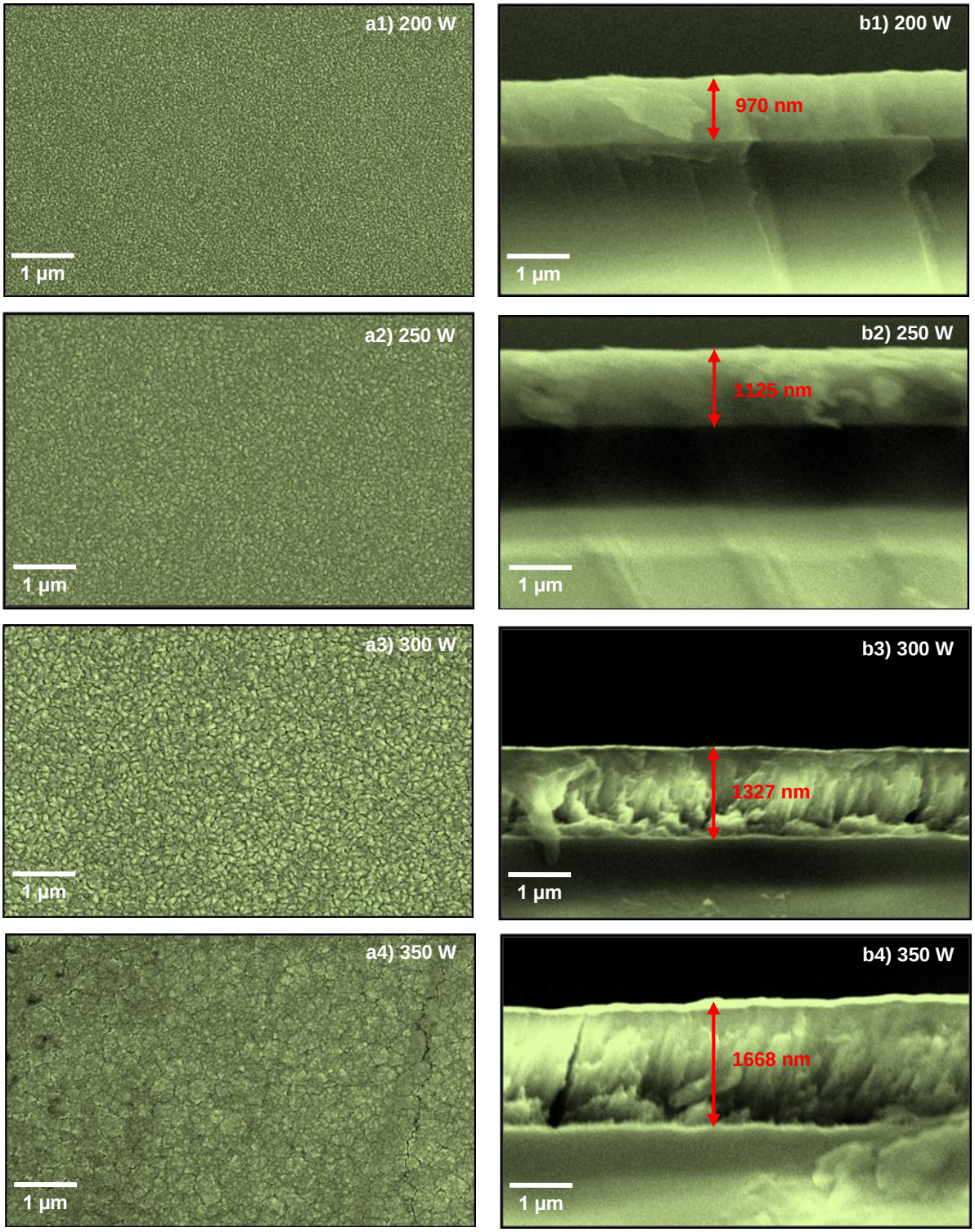

Fig. 3 – FE-SEM micrographs of Mo thin films deposited at different DC sputtering powers a1-a4) Surface morphology and b1-b4) Cross-section view of FE-SEM micrographs

3.4 FE-SEM Analysis

The surface morphology of sputtered Mo thin films deposited at different DC sputtering powers has been investigated by using scanning Fourier transform electron microscopy (FE-SEM). Figure 3(a1)-(a4) shows FE-SEM micrographs of Mo thin films deposited at high DC sputtering power.

As seen except the Mo film deposited at DC sputtering power of 350 W, all films are dense, homogeneous and free of flaws and cracks. The FE-SEM images clearly show tiny irregular prismatic shape like morphology with increasing particles of size. The Mo film deposited at DC sputtering power of 350 W shows some cracks and fractures. Appearance of cracks and fractures are due to compressive strain in the film. Low angle XRD analysis further supports this. Figure 3(b1)-(b4) shows cross-section of FE-SEM micrographs of Mo thin films deposited at high DC sputtering power used for the thickness measurements. Thickness measurement done by cross-section micrographs is well matched with thickness measured by KLA Tencor, P-16+ profilometer.

3.5 Atomic Force Microscopy (AFM) Analysis

The surface topography of DC-Magnetron sputtered Mo thin film was studied by non-contact atomic force microscopy (NC-AFM). The surface properties of sputtered Mo films are investigated by measuring root mean square (RMS) roughness values. Fig. 4 represents the 3-D AFM images of Mo thin films at deposited at various DC sputtering powers.

It is observed that the surface roughness of the films increases from 14.70 nm to 23.24 nm when DC sputtering power increased from 200 W to 350 W.

3.6 UV-Visible Spectroscopy Analysis

The optical properties of back metal contact Mo thin films play an important role in the efficiency of CZTS and CIGS solar cells. In order to understand the optical reflection properties of DC-Magnetron sputtered Mo thin films, the reflectance of Mo films deposited at different DC sputtering power was recorded using a UV-Visible spectrophotometer. The results are plotted in Fig. 5 as a function of wavelength. As seen from the figure, the average reflection of Mo thin films in the visible range (400 to 800 nm) increases from ~ 16 to ~ 23 %, when the DC sputtering power increases from 200 W to 350 W.

The increase in reflection may be due to the increase in average grain size and surface roughness with increase in DC sputtering power. The increase in average grain size and surface roughness enhances optical scattering. The XRD and NC-AFM analysis supports this conjecture. It has been reported that the existence of a thin oxide layer on the Mo surface plays an important role in the determination of the reflectance of Mo back contacts [14]. Therefore, apart from the increase in average grain size and surface roughness with increase in DC sputtering power the presence of a thin oxide layer of Mo (MoO_x) on the Mo surface may also affect the reflection. However, the study related to presence of MoO_x thin layer over the Mo surface is beyond the scope of the present work.

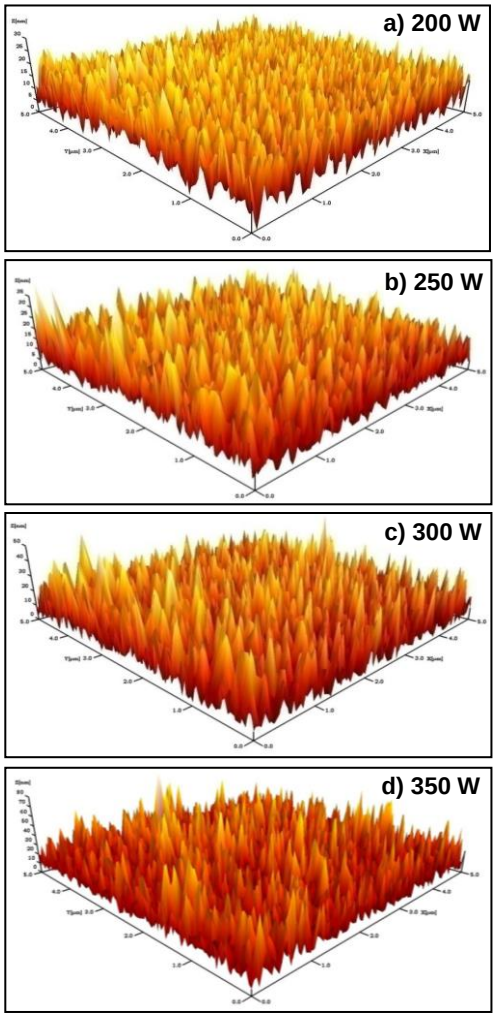

Fig. 4 – 3-D AFM images of Mo thin films at deposited at various DC sputtering powers a) 200 W b) 250 W c) 300 W and d) 350 W

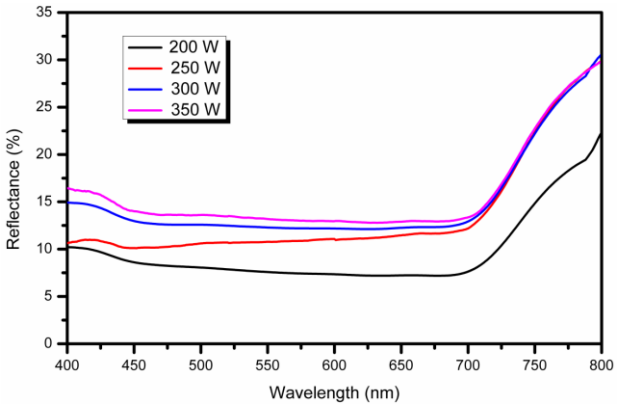

Fig. 5 – VU-Visible reflectance spectra of Mo films deposited at different DC sputtering powers

3.7 Electrical properties

The Mo back contact with low electrical resistivity is essential to minimize the series resistance of solar cell device. Hall Effect study is a powerful tool for knowing the electronic properties of thin film samples. Electrical resistivity, carrier mobility and carrier con-

centration of Mo films was measured using Van der Pauw method (Ecopia HMS-3000 Hall Measurement System) at room temperature. For this, four contacts were made by using indium wire at the four corners of the film sample of dimension of 1 cm×1 cm. The gold spring probes were placed at the corners of the sample symmetrically. The films were subjected to uniform magnetic field of 0.54 Tesla. A known current (1μA) was passed through the electrodes of the films, and voltage across the other two electrodes was measured. Fig. 6 shows the variation of electrical resistivity, carrier mobility and carrier concentration conductivity of Mo thin films deposited at different DC sputtering powers.

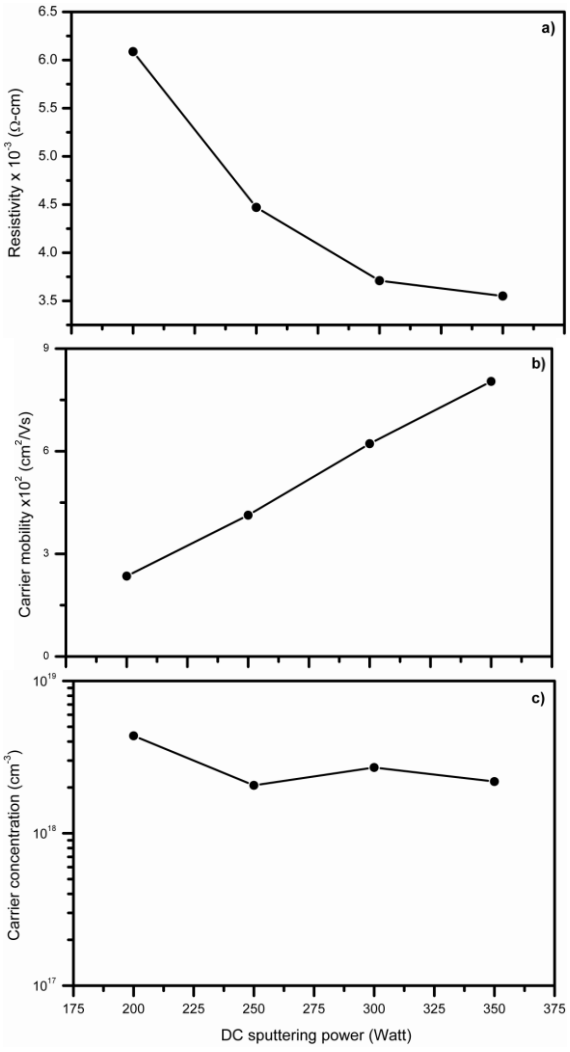

Fig. 6 – Electrical properties of Mo films deposited at different DC sputtering powers a) Electrical resistivity b) Charge carrier mobility and c) Charge carrier concentration

As seen from the figure, the electrical resistivity gradually decreases from ~ $6.09{\cdot}10^{-3}$ Ω·cm to ~ $3.55{\cdot}10^{-3}$ Ω·cm when the DC sputtering power increased from 200 W to 350 W (Fig. 6(a)). The mobility of charge carrier shows increasing trend with increasing DC sputtering power. It increases from ~ 235 cm²/Vs to ~ 750 cm²/Vs when the DC sputtering power increased from 200 W to 350 W (Fig. 6(b)). However, the charge carrier density almost remains constant in the range of ~ 10^{18}-10^{19} A/cm³ (Fig. 6(c)) over the entire range of DC sputtering power studied. The decrease in electrical resistivity with increase in DC sputtering power can attribute to increasing grain size of Mo thin film with increase in DC sputtering power. A larger grain size decreases the scattering of charge carriers and grain boundary, which enhances the carrier lifetime and, consequently, the decrease in electrical resistivity is observed [13].

4. CONCULSIONS

In conclusions, we analyzed the effect of the DC sputtering power on structural, electrical, optical and morphological properties of Mo thin films deposited by DC-Magnetron sputtering method. Cross-hatch cut and Scotch tape adhesion test showed that all Mo thin films have good adhesion to glass substrate. The XRD analysis showed that as-deposited Mo films have preferred orientation in (110) direction and with increase in DC sputtering power enhancement in its crystallinity and average grain size were observed. Furthermore, XRD analysis showed that the Mo films deposited at DC sputtering power < 300 W exhibit tensile strain and its value decreases with increase in DC sputtering power, while the Mo film deposited at DC sputtering power > 350 W exhibit compressive strain. Surface morphology investigated using FE-SEM showed that except DC sputtering power of 350 W, all Mo films are dense, homogeneous and free of flaws and cracks. Non-contact atomic force microscopy (NC-AFM) revealed that the surface roughness of the films increases from 14.70 nm to 23.24 nm when DC sputtering power increased from 200 W to 350 W. The UV-Visible spectroscopy analysis showed that an average reflection of Mo films in the range of ~ 16-23 % in the visible range of the spectrum. The electrical properties carried out using Hall Effect measurements showed that resistivity of Mo films decreases with increase in DC sputtering power whereas charge carrier mobility shows increasing trend. On the other hand, the charge carrier density remains almost constant (~ 10^{18}-10^{19} A/cm³) over the entire range of DC sputtering power studied. The obtained results suggest that as-synthesized Mo thin films with DC power < 300 W have potential application as a back contact material for CZTS thin film solar cells due to good adhesion and low electrical resistivity.

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Магнетронне розпилення при постійному тоці зворотного контакту Mo для тонкоплівкових сонячних елементів з халькопіриту

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В даній роботі, плівки Mo осаджувалися на підкладках із скла з використанням магнетронного розпилення при постійному струмі. Досліджено вплив потужності розпилення на електричні, структурні, морфологічні, оптичні та топологічні властивості за допомогою ефекту Холла, рентгенівської дифракції, автоелектронної скануючої мікроскопії, спектроскопії в УФ та видимої областях, неконтактної атомно-силової мікроскопії, тощо. Виявлено, що потужність постійного струму суттєво впливає на електричний опір і адгезію тонкої плівки Mo. Синтезовані плівки Mo мали товщину декількох мікрометрів (~ 0.9-1.6 мкм) зі швидкістю осадження в діапазоні 32-57 нм/хв. Випробування показали, що всі плівки Mo мають гарну адгезію до підкладки. Рентгено-дифракційний аналіз показав, що свіжосконденсовані плівки Mo мають переважну орієнтацію (110) і поліпшення її кристалічності та середнього розміру зерна зі збільшенням потужності розпилення при постійному струмі. Крім того, рентгенодифракційний аналіз показав, що плівки Mo, нанесені при потужності розпилення < 300 Вт, демонструють деформацію розтягування, в той час як нанесені при потужності розпилення > 350 Вт демонструють деформацію стиску. Результати автоелектронної скануючої мікроскопії показали, що всі плівки Mo щільні, однорідні і вільні від дефектів і тріщин. При спектроскопічному аналізі спостерігалося збільшення середнього коефіцієнту відбиття плівок Mo з потужністю розпилення спостерігалося у видимому діапазоні спектра. Неконтактна атомно-силова мікроскопія показала, що шорсткість поверхні плівок збільшується зі збільшенням потужності розпилення при постійному струмі. Вимірювання ефекту Холла показало, що електричний опір плівок Mo зменшується, а рухливість носіїв заряду збільшується з ростом потужності розпилення при постійному струмі. Отримані результати свідчать про те, що синтезовані тонкі плівки Mo з потужністю постійного струму < 300 Вт мають перспективу застосування як матеріалу зворотного контакту для сполук халькопіритів на основі сонячних елементів завдяки хорошій адгезії та низькому електричному опору.

Ключові слова: Молібден, Опір, Розпилення при постійному тоці, Атомно силова мікроскопія, Рентгенівська дифракція.