Synthesis of Al Thin Films with High Optical Transmittance by DC Magnetron Sputtering Process Parameter Optimization

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***Abstract* — Nanostructured Al thin film with higher optical transmittance and electrical conductivity has intensive applications in solar cells and optical and microelectronic devices. This experimental-based research study has optimized the DC magnetron sputtering deposition parameters (sputtering power, sputtering current, voltage, and working gas pressure) for Al thin film deposition to obtain the highest optical transmittance and lower sheet resistance. Optical transmittance, surface roughness, film thickness, sheet resistance, grain size, and surface morphology were characterized using UV-vis-NIR spectroscopy, surface profiler, spectroscopic ellipsometry, four-point probe, and FE-SEM, respectively to determine the effects of sputtering process parameters on Al films' different properties. Experimental investigations reveal that electrical conductivity, surface roughness, grain size, and deposition rate increase with increasing of sputtering power at certain working gas pressure. At the optimized condition (sputtering power 80 W, working gas pressure 5 mTorr, deposition time 5 min and ambient temperature), the relatively higher optical transmittance in visible region 96%, moderate sheet resistance 0.196 Ω/□ and lowest average surface roughness 2.86 nm were obtained for Al thin film. After all, this research study will help to understand the best Al film deposition parameters in terms of optical transmittance and electrical conductivity for future research and industrial applications.**

***Keywords* — Al Thin Film, DC Magnetron Sputtering, Optical Transmittance, Sheet Resistance, Surface Roughness.**

I. Introduction

To date flexible device fabrication technology in micro-electronics, optoelectronic, and thin film solar cells-based devices has attracted huge attention for its lightweight, easy to fold, and lower fabrication cost [1]-[13]. Aluminum (Al) thin film is widely used in flexible device fabrication technology due to its superior electrical, optical, and thermal properties as ohmic contacts, Schottky contacts, gate electrodes, back contacts, interconnects, and good light reflectors [5], [7], [10], [11], [13]. Al thin films can be deposited on glass and polymer substrates to facilitate the fabrication of optical and microelectronic devices using DC (direct current)/RF (radio frequency) magnetron sputtering, thermal evaporation, and electron beam evaporation (e-beam) techniques [3], [4], [9], [14]-[17]. In comparison with other existing deposition techniques, DC magnetron sputtering-based Al thin film deposition on various substrates is widely adopted due to the excellent thin film properties and repeatability with optimum controllability. However, the micro-structural characteristics (morphology, grain size, density, and textures) strongly affect the electrical, optical, functional, and structural properties of Al thin films, which can drastically reduce the performance of flexible devices [3], [5], [8]-[13], [15], [18]-[22].

Therefore, it is very important to understand the influence of Al thin film deposition process parameters (sputtering power, sputtering pressure, deposition rate, and thin film thickness) on its micro-structural characteristics to enhance the functionality of Al thin films in different devices fabrication. A few researchers have conducted studies for Al thin film deposition on glass and polymer substrates using e-beam or thermal evaporation techniques and few researchers have reported Al thin film deposition process parameters on glass substrates using DC magnetron sputtering [5], [7], [10], [13]. However, the reported results to fully understand the DC magnetron-based Al thin film deposition process parameters are still in the infancy phase. Al thin films with the lowest resistivity, maximum optical reflectivity, higher compactness, and homogeneity are the key factors to boost the optical, microelectronic, and solar energy devices at elevated levels [10]-[13], [16], [17]. As a consequence, further systematic study to clarify each influential deposition process parameter on the micro-structural, electrical, and optical properties of Al thin films is very necessary to maximize the performance of microelectronic and optical devices with lower fabrication costs.

In this systematic research study, Al thin films were deposited on glass substrates by DC magnetron sputtering technology at room temperature. In order to optimize the Al thin films' deposition process parameters, the effects of process parameters (sputtering power, sputtering current, sputtering voltage, working pressure, and sputtering rate) on the electrical, optical, and micro-structural properties have been studied in detail.

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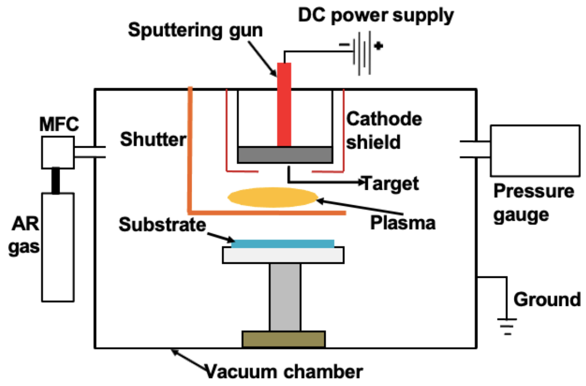


Fig. 1. Schematic diagram of DC magnetron sputtering technique for Al thin film deposition.

II. Experimental Details

All Al thin films were deposited on glass (microscope slides) substrates by DC magnetron sputtering technique at room temperature using a 99.99% pure Al target (50.8 mm diameter and 6.35 mm thickness). The schematic diagram of DC magnetron sputtering technique for thin film deposition is shown in Fig. 1. The glass substrates (2 cm × 2 cm) were cleaned ultrasonically in an acetone and ethanol bath and dried under nitrogen gas. The chamber base pressure was 2 × 10−6 Torr and the sputtering was conducted in Argon (99.99% purity) gas atmosphere, and the target-substrate distance was fixed at 5 cm through the whole deposition process. The thin film deposition time was fixed (5 min) while sputtering power and working gas pressure was 80 ~ 200 W and 3.5 ~ 10 mTorr, respectively. To avoid contamination, the Al target was pre-sputtered for 10 min prior to actual Al thin film deposition with a shutter located in between the target and substrate.

The thin film thickness was measured using a state-of- the-art nondestructive spectroscopic ellipsometry (K-MAC ST4000-DLX) technique based on the constructive and de-structive interference principle in the spectrum of white light incident on the surface of the Al thin film. The average surface roughness (Ra) of Al thin film was investigated by a NanoView high accuracy 3D nano noncontact surface profiler system (non-contact white light scanning interferometer system, WT-250) at 20× objective. The surface morphology of Al thin films was characterized by a field emission scanning electron microscope (FE-SEM, JSM-6700F) with an acceleration voltage of 15 KV, and the grain size of different sputtering power deposited films was also characterized with the FE-SEM technique. The optical transmittance and sheet resistance (electrical conductivity) of the Al thin films de- posited on glass substrates were measured using a UV-vis-NIR spectrophotometer (Shimadzu UV-3150) and four-point probe respectively to analyze the effects of process parameters on optical and electrical properties.

III. Results and Discussion

A. Sputtering Power and Pressure Effects on Al Target Sputtering Rate

The sputtering power is very closely linked to working gas pressure for the deposition of different thin films according to theory and experimental observations of magnetron sputtering [4], [5], [11]-[13], [15]-[19], [22]. The selective selection of magnetron sputtered thin film/composite films' deposition conditions is a very crucial issue to get optimum physical, electrical, and chemical properties [4], [16], [17]. In the case of DC magnetron sputtered-based thin film deposition, the sputtering rate, working gas pressure, sputtering power, and current are very important, which control the deposited thin film properties significantly.

According to a previous magnetron sputtered-based thin film study, an increase in sputtering/deposition power increases the sputtering rate due to higher sputtering yield and current density, which finally increases the thin film thickness for the same deposition time. The sputtering rate of a target material for low to medium ion energy can be calculated using (1) [4].

$$\frac{dN}{Adt} = Y\frac{I}{q} \quad (1)$$

In (1), the total number of sputtered atoms is N, the area of the target material (cathode surface) is A, the yield of sputtering is Y, the plasma ion current is I and q is the electronic charge of ions. The relationship between sputtering rate and other relevant experimental plasma-based deposition parameters (working gas pressure, distance between cathode to anode, and flux of sputtered particles) can be also explained by Keller-Simmons (K-S) formula [12]. According to the K-S formula and (1), the sputtering rate increases if the ionized and sputtered particles become more energetic relying on higher sputtering power.

For a specific lower sputtering power, the higher working gas pressure usually makes the sputtering atoms less energetic that resulting in decreasing in sputtering rate, and the substrate surface inside the magnetron sputtering technique is covered with charged particles according to magnetron sputtering theory [4], [12], [16]. If the sputtering power is kept constant, an initial increase in working gas pressure could increase the sputtering rate due to the increase in ion density by following (1). The sputtering rate was increased with the increase of working gas pressure from 3.5 to 7.5 mTorr for all sputtering powers in the range of 80 ~ 140 W shown in Fig. 2, which may be due to the consecutive increase in ions density. In addition, the sputtering rate was also increased with the increase of sputtering power (80 ~ 200 W) as the higher sputtering power gives higher sputtering yield and current density according to (1) and K-S formula [4], [12].

The further increase in working gas pressure after a certain value (saturation point) for the constant sputtering power could reduce the sputtering rate due to the back diffusion and collision between sputtered particles and chamber particles (ions and argon gas) [16]. If the geometry of the magnetron sputtering technique is fixed, the saturation points of working gas pressure varies depending on different sputtering power as the sputtering yield, ions current, ion density, particle collision, and kinetic energy of sputtered atoms are changed according to magnetron sputtering theory. The sputtering rate decreasing trend due to back diffusion and particle collisions was noticed for sputtering power 80, 100, 120, and 140 W with working gas pressure 10 mTorr shown in Fig. 2.

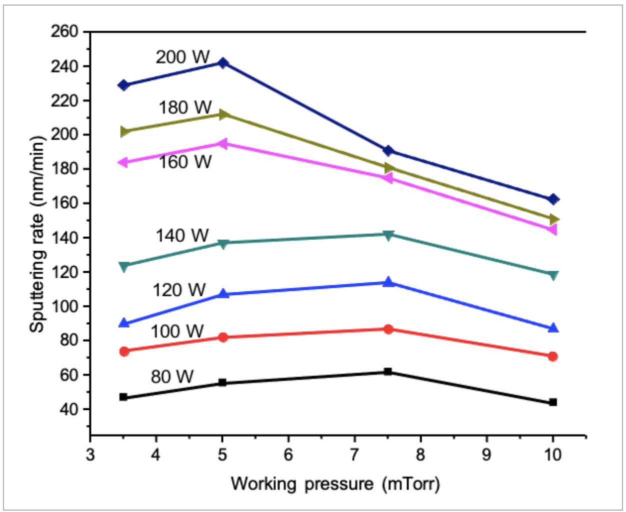


Fig. 2. DC magnetron sputtering rate of Al target at different sputtering power with the variation of working gas pressure.

On the other hand, the drastic fall in the sputtering rate for higher sputtering or deposition power (160, 180, and 200 W) was observed at lower working gas pressure (after 5 mTorr) compared with the lower sputtering power (80, 100, 120 and 140 W), because the saturation point of working gas pressure was reached comparatively early for higher sputtering power according to magnetron sputtering theory [4]. For constant sputtering power, the sputtering rate decreases, if the working gas pressure is very high because the ionized particles are not sufficiently energetic to sputter the target material as the particle's mean free path, is less. The experimental findings from the conducted study to understand the variation in sputtering rate depending on various sputtering power and working gas pressure effects are well agreed with previously published magnetron sputtering and thin film deposition theory.

B. Variation in DC Magnetron Sputtering Current and Voltage

According to the plasma discharge theory in particle and gas ionization, the sputtering current and voltage play a very important role along with sputtering power and working gas pressure to control the sputtering rate and properties of magnetron-sputtered thin films [3]-[5], [16], [23]. Based on previous empirical and theoretical studies, the sputtering rate increases with the increase of sputtering current due to the higher flow of ionized particles towards the target cathode as the sputtering yield relatively increases. In Fig. 3, the sputtering current increases with the increase of sputtering power (80 ~ 200 W) and working gas pressure (3.5 ~ 10mTorr). For any sputtering power shown in Fig. 3, the current increases with the increase of working gas pressure because argon gas density is increased in the magnetron sputtering chamber which results in more particles available to move towards the sputtering target (cathode) [23]. The increase in sputtering current for all the sputtering powers contributes to increasing in sputtering rate finally, which can be noticed in Fig. 2 and Fig. 3 as a comparison.

In contrast, the sputtering voltage decreases for any sputtering power shown in Fig. 4 with the increase of working gas pressure, which is quite opposite to the sputtering current increasing trend. The relationship between sputtering voltage (V) and current (I) at a constant sputtering power (P) with the variation of working gas pressure can be calculated using (2).

$$I = \frac{P}{V} \tag{2}$$

The sputtering rate increases slowly with the increase of sputtering voltage, as the energy of ionized particles is increased slowly. However, most of the previous studies with regard to magnetron-based thin film deposition suggest using sputtering current as a basic deposition parameter as voltage plays less role to increase sputtering yield in comparison with the sputtering current.

At a certain stage with specific sputtering conditions, the increase in voltage with the increase of sputtering power (160, 180, and 200 W) becomes very slow compared to lower sputtering power (80, 100, 120, and 140 W) shown in Fig. 4 and all the supplied energy due to the increase of sputtering power is used to increase the sputtering current value only [5], [16], [23]. In Fig. 4, it is clearly seen that the increase in sputtering power from 160 to 200 W has very less impact on the increase of sputtering voltage value. Therefore, this experimental study as well as previous magnetron sputtering observations suggests using sputtering current instead of sputtering voltage as the basic deposition parameter for DC magnetron-based thin film deposition.

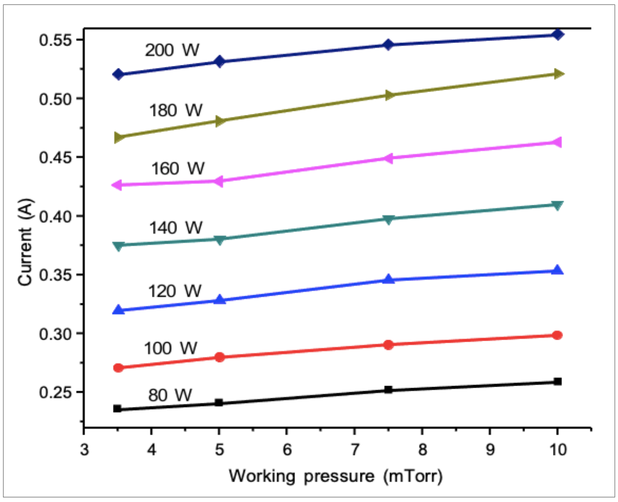


Fig. 3. Working gas pressure effect on DC magnetron sputtering current at various sputtering powers.

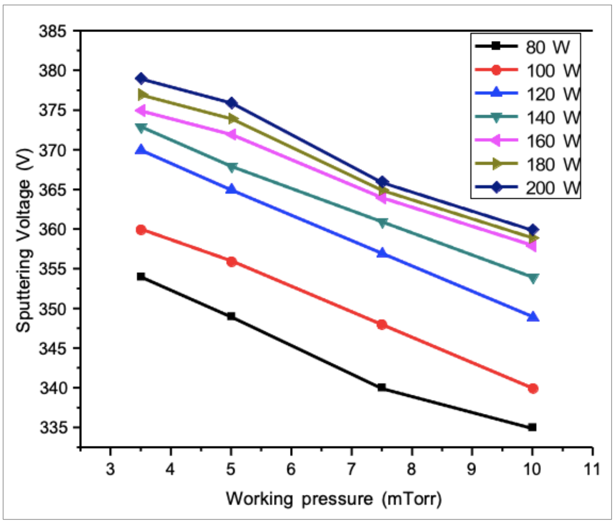


Fig. 4. Working gas pressure effect on magnetron sputtering voltage at various sputtering powers.

C. Optimal and Surface Properties

Aluminum (Al) thin film is widely used as back contacts, good light reflectors, and interconnectors in flexible device fabrication technology, so the overall transmittance of used Al films would significantly affect the device performance, especially in the case of solar cell applications [7], [10], [11], [13], [22]. As a result, the effects of deposition powers (80, 100, 120, and 140 W) at a constant working gas pressure (5 mTorr) on Al film's overall transmittance were analyzed with a UV-vis-NIR spectrometer shown in Fig. 5. The average optical transmittance in the visible wavelength region 400 ~ 800 nm of deposited Al films at 80 W and 100 W deposition power is around 96% and 88%, respectively. However, the average transmittance in the wavelength region 400 ~ 800 nm decreases from 96% to 73% with the increase of deposition power from 80 W to 140 W, which might be due to the increase in Al film thickness, crystal growth, sputtering rate, and grain size [3], [7], [9], [11]-[13], [18], [20], [22], [23]. The higher deposition power usually increases the adatom mobility, sputtering yield, and defects in crystal growth according to magnetron sputtering theory and experimental observations, which contribute to reducing the optical transmittance.

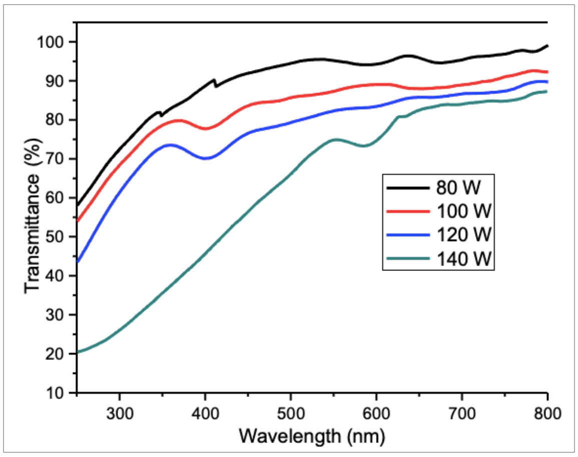


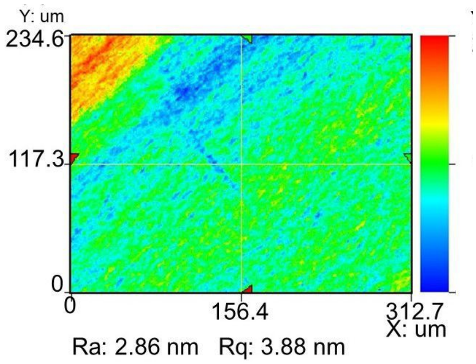
Fig. 5. Optical transmittance spectra of deposited Al films with various deposition powers at constant working gas pressure (5mTorr).

The increase in film thickness, average surface roughness, and grain size with the increase in deposition power is shown in Table I.

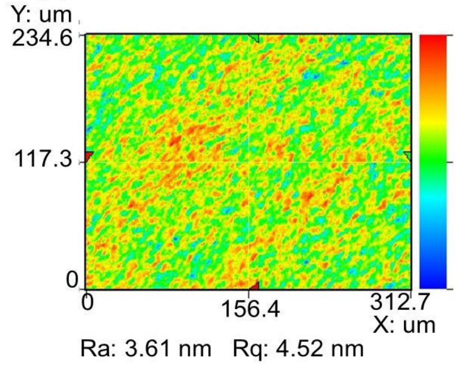
TABLE I: DEPOSITION PARAMETERS INFLUENCE ON AL FILM THICKNESS (NM), AVERAGE SURFACE ROUGHNESS (R_A IN NM), AND GRAIN SIZE (NM)

Parameters	Size (nm)	R_a (nm)	Thickness (nm)
80 Watt (5 mTorr)	131.4	2.86	276.97
80 Watt (7.5 mTorr)	176.5	3.03	309.1
100 Watt (5 mTorr)	202.7	3.61	410.7
120 Watt (5 mTorr)	238.33	7.06	535.3
140 (5 mTorr)	257.5	8.81	685.4
180 (5 mTorr)	319.5	9.85	1060
200 (5 mTorr)	542.6	11.91	1210

The lowest average surface roughness 2.86 nm shown in Fig. 6(a) and grain size of 131.4 nm are observed for lower deposition power of 80 W. The surface roughness and grain size were 3.61 and 202.7 nm respectively, for 100 W deposition power shown in Fig 6(b) and Table I. Furthermore, the highest deposition power 200 W increased the surface roughness and grain size to 11.91 and 542.6 nm, respectively.



(a)



(b)

Fig. 6. 2D surface profile of DC magnetron sputtered Al thin films: a) 80 W; b) 100 W deposition powers.

The grain size (131.4 nm 542.6 nm) and average surface roughness (2.86 ~ 11.91 nm) of Al films increase with the increase of deposition power from 80 W to 200 W respectively, as the higher deposition power increases the adatom mobility and sputtering rate significantly at a certain deposition condition.

It is clearly seen from Table I that the average surface roughness and grain size of Al films with deposition power 80 W increased with the increase in working gas pressure from 5 mTorr to 7.5 mTorr.

The effect of sputtering pressure on surface roughness and grain size can be explained by (3) [9].

$$Mean\ free\ path = 2.330 \times 10^{-20} \times \frac{T}{W_p \times d^2} \quad (3)$$

Here, W_p denotes working gas pressure and d denotes molecular diameter. According to (3), the mean free path of sputtered atoms in a magnetron sputtering chamber at higher working gas pressure (W_p) is reduced compared to less working gas pressure at constant sputtering power. As a consequence, the sputtered atoms at higher working gas pressure undergo many collisions resulting in a higher probability of agglomeration, and the higher probability of agglomeration increases the particle size before arriving at the substrate surface which finally could increase the Al film surface roughness [4], [23], [15].

D. Electrical Properties

Due to the numerous applications of Al films in micro-electronic devices such as back contacts, reflectors, interconnectors, and so on, the sheet resistance is a very important parameter of Al films to maximize device performance [7], [10], [11], [23]. To understand the sputtering power effects on sheet resistance (Ω/□) in parallel with film thickness, a four- point probe-based Al films' sheet resistance measurement was conducted shown in Fig. 7. The sheet resistance was 0.196 Ω/□ at deposition power 80 W when the thickness of Al film was 276.97 nm; however, the sheet resistance decreased rapidly with the increase of deposition power as shown in Fig. 7. The minimum sheet resistance and highest film thickness were 0.068 Ω/□ and 1210 nm respectively at deposition power 200 W. According to magnetron sputtering theory and previous discussion, higher deposition power usually increases adatoms mobility, deposition rate, uniformity of the film, crystal quality, and grain size, which could contribute to decreasing the Al film sheet resistance [4], [5], [9], [12], [23]. Therefore, it can be concluded that the sheet resistance is decreased with the increase in film thickness at a certain limit.

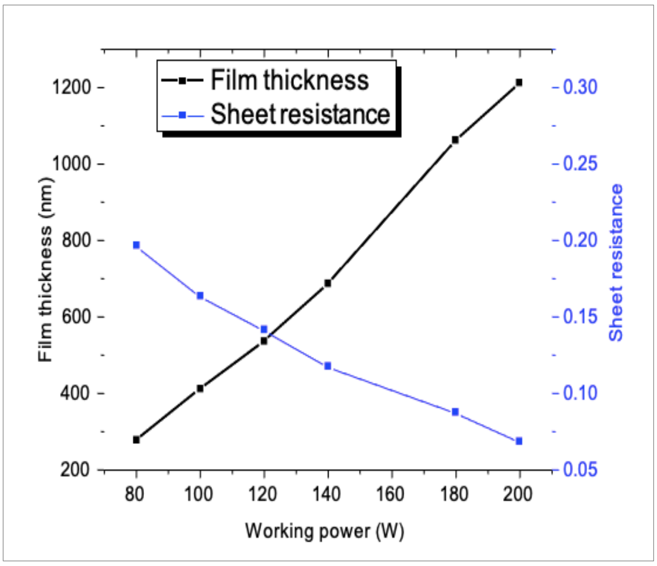


Fig. 7. Variations of Al films thickness and sheet resistance as a function of deposition power at 5 mTorr.

E. *Surface Morphology and Structural Characterization*

The FE-SEM is a well-established technique to analyze sur- face morphology for better insights into surface structure, grain size, roughness, and defects [24]-[28]. The FE-SEM images of Al films deposited at varying sputtering power (80 W, 100 W, and 200 W) are shown in Fig. 8(a-c). The adatom mobility, grain size and surface roughness exhibit an increasing trend with the increase in sputtering power shown in Fig. 8(a-c), because the higher deposition power increases the sputtering yield, the kinetic energy of sputtered atoms and ions current [9], [23]. The increasing trend in surface roughness with the increase in sputtering power is also confirmed by the 3D nano surface profiler measurement reported in Table I.

The Al film with deposition power 80 W shows uniform surface morphology with compact fine crystal grain. The grain size and density of the film increase with the increase in deposition power from 80 W to 200 W which is clearly visible in Fig. 8(a-c).

To verify the grain size and crystal quality of deposited Al thin films, the structures of Al films deposited at 80 W and 100 W were analyzed using a Rigaku D-Max 2400 x-ray diffractometer with Cu Kα radiation operating at 40kV, 40mA shown in Fig. 9(a-b).

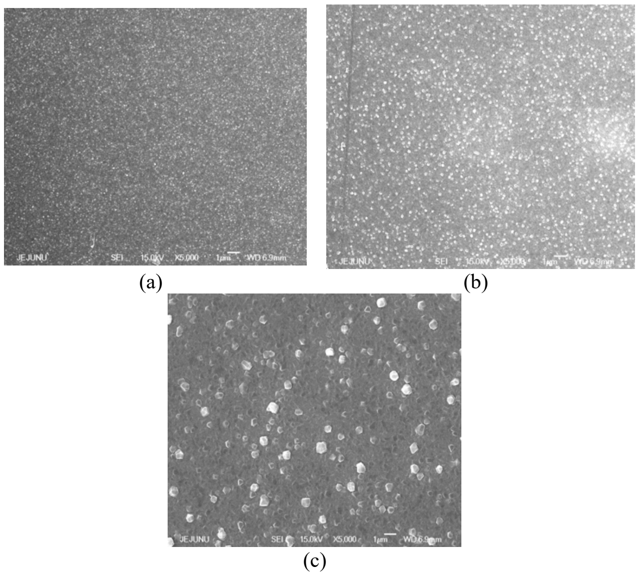


Fig. 8. FE-SEM images of Al films to understand surface morphology with different deposition powers at 5 mTorr: a) 80 W; b) 100 W; c) 200 W.

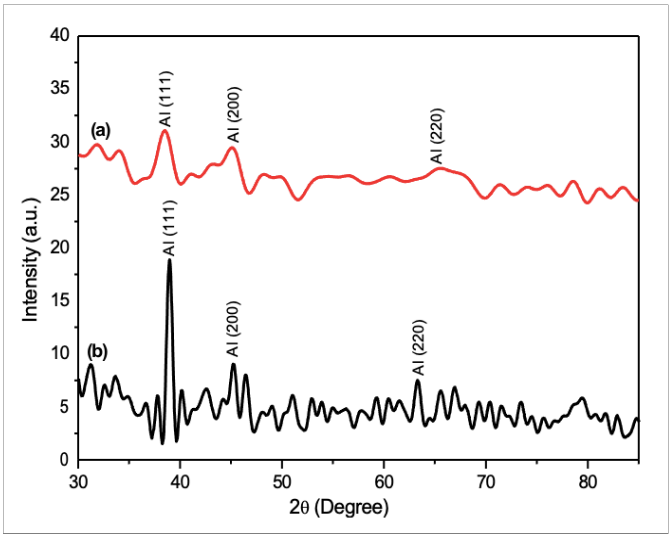


Fig. 9. XRD patterns of Al thin films deposited with different sputtering power at 5 mTorr working gas pressure: (a) 80 W and (b) 100 W.

In Fig. 9(a), the 2θ peak appeared at 38.52, 45.10, and 65.14 degrees with the orientation of 111, 200, and 220 respectively corresponding to Al crystal peaks (JCPDS number: 00-004-0787) [21], [29].

The intensity of Al peaks at 38.520 (111), 45.100 (200), and 65.140 (220) was increased with the increase of Al thin film deposition power shown in Fig. 9(a-b).

In addition, the increase in deposition power also decreased the width of Al peaks. The increase in Al peak intensity and decrease in peak width with increasing power denotes that the crustal quality and grain size of deposited Al thin film at 100 W deposition power was increased compared to lower deposition power (80 W). The increasing trend of crystal quality and grain size with the increase of deposition power from XRD observations is also well agreed with SEM observations reported in Table I and Fig. 8.

Therefore, it can be stated that adatom mobility, deposition rate, uniformity of the film, crystal quality, and grain size usually increase with the increase of deposition power according to the conducted experimental observations and magnetron sputtering theory.

IV. Conclusion

In this research study, with comparatively higher optical transmittance and lower sheet resistance, Al thin films on glass substrates were deposited using DC magnetron sputtering technique at ambient temperature. The sputtering process parameters (sputtering current, voltage, and working gas pressure) for Al thin films deposition at various sputtering powers have been studied, and their effects on deposited Al thin films properties (transmittance, surface roughness, morphological sheet resistance, grain size, and deposition rate) are discussed in detail.

According to systemic experimental investigations, optimal properties of Al thin film were obtained with sputtering power of 80 W, sputtering pressure of 5 mTorr, and sputtering time of 5 min at ambient temperature compared with other sputtering power and working gas pressure. At the optimized conditions, the transmittance, sheet resistance, grain size, thickness, and deposition rate of Al film were 96%, 0.196 Ω/□, 131.4 nm, 276.97 nm, and 55.4 nm/min, respectively. In addition, the surface morphology was uniform with a decent crystal grain. Our studies also found that electrical conductivity, grain size, deposition rate, and surface roughness increase with increasing sputtering power as higher sputtering power increases sputtering yield, adatom mobility, and kinetic energy of sputtered atoms. Moreover, the experimental findings of this research study with regard to the effects of sputtering process parameters on Al thin film properties are aligned with previously published magnetron sputtering theory and experimental investigations.

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Conflict of Interest

Authors declare that they do not have any conflict of interest.

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