

Influence of sputtering power on surface topography, microstructure and mechanical properties of aluminum thin films

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Abstract—The article reports on the influence of sputtering power on surface topography, microstructure and mechanical properties of aluminum thin films sputtered on mild steel substrates. The Al thin films were deposited on mild steel substrate at varying RF power of 150 W, 250 W, 300 W, and 350 W and at a constant substrate temperature of 90°. The films were characterized by optical surface profiler (OSP), field emission scanning electron microscopy (FESEM) and nanoindentation. The highest average and root mean square roughness values were obtained at 250 W while the lowest at 350 W. The highest and smallest Rt values were obtained at 150 W and 350 W respectively. The lowest and highest Rp values were determined at 350 W and 250 W respectively. The lowest hardness and modulus were obtained at 250 W while the highest values were determined on films sputtered at 150 W. The FESEM micrographs revealed that at 150 W the structure consisted of very fine and interconnected oxides and porosity whereas, at 350 W, they exhibited circular and well-defined secondary grains. These observations were discussed in relation to the magnetron sputtering process and RF power.

Keywords—Aluminum; sputtering; nanoindentation; power; a substrate; thin films

I. Introduction

The surface roughness, microstructure, and nanoindentation properties significantly influence the performance and hence the application of aluminum thin films [1]. These properties depend on the type of deposition method and substrate, deposition parameters and conditions. There are various methods, which have been used to prepare aluminum thin films some of which include chemical vapor deposition (CVD), thermal spray, sputtering, etc. [2]. Although CVD methods have been widely used, they are mostly undertaken at high-temperature conditions and therefore difficult to control. Thermal spray methods have also been used especially for thick coatings of Al films, however, they are associated with the formation of highly porous structures [3].

Sputtering technique is a physical vapor deposition (PVD) technique which is easy to tune and hence control the properties of the formed films.

The effect of various sputtering parameters on the properties of Al thin films has been reported [1]. The effect of substrate type on the microstructure and roughness of Al on glass, wafer and steel substrates was studied and reported that depositing on steel results in the formation of the large-grained microstructure of Al thin films [4]. Al thin films deposited on stainless steel and mild steel substrates via magnetron sputtering were shown to exhibit different topography and microstructures [5]. The state and type of the target on DC magnetron sputtering of Al films have been studied [6]. The effect of substrate temperature on the properties of Al thin films deposited through magnetron sputtering has also been reported [7], [8]. The deposition rate (film's thickness and time) and temperature have also been shown to affect the sputtering of Al films [9]–[11]. The effect of argon gas pressure on Ti, Al and TiAl thin films has been reported [12].

The sputtering power has been shown to influence the properties of thin films such as TiO₂ [13], [14], Ca-doped ZnO [15], Cu films [16], Bi₂Se₃ [17], ITO films [18], Al-doped ZnO [19], ZnS films [20], Cu_xAl_yO_z films [21] and so forth. In all these reports, various films behave differently under varying power and there is no proportional relationship between the surface properties and the increase in sputtering power. There are limited studies on the effect of power on the properties of Al thin films. The purpose of this work, therefore, is to study the evolution of microstructure, surface roughness and nanoindentation of Al thin films with sputtering powers. The work can be useful for industry in optimizing the quality of sputtered Al films for applications in microelectronics and optical devices.

II. Methods

A. Magnetron sputtering

Pure aluminum (99.99%) targets of 75 mm diameter and 3 mm thickness mounted on copper backplates were used. Mild steel plates of 50 mm by 50 mm by 2 mm were used as the substrates. Before sputtering, the mild steel substrates were ground to remove the oxide layers, degreased in acetone for 3-5 minutes and then hot-air dried. The substrate and target were mounted at a fixed distance of 13 cm. The deposition chamber was kept at a vacuum pressure of 1×10^{-5} mbar. The pre-sputtering was undertaken, with the target shutter closed, for 10 minutes to further clean the substrate and target surfaces.

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The substrate holder rotation was kept at 5 rpm, argon flow rate at 12.0 sccm and the substrate temperature at 90°C. The deposition was then conducted at a power range of 150-350 W. Further details of the RF magnetron sputtering and parameters are described elsewhere [5].

B. Characterizations

The samples were then sliced into 10 mm by 10 mm for surface property characterizations. The topography was undertaken by Contour Elite K non-contact optical surface profiler (OSP) manufactured by Bruker (USA). The microstructure was obtained by Carl ZEISS Sigma FESEM (Germany). The nanoindentation was undertaken by TI 950 TriboIndenter™ nanomechanical (Hysitron, Inc., USA) through load-control method. The nanoindentation was undertaken at a maximum load of 250 mN.

III. Results and Discussions

Figs. 1 and 2 are the representative optical surface profiler images of the Al thin films deposited at 150, 250, 300 and 350 W. The profiling parameters are summarized in Table I. In the table, average roughness (Ra), root mean square roughness (Rq), distance between the highest peak and lowest valley (Rt) and the highest point of the profile above the mean line (Rp) within the assessment length were used to characterize the surface roughness. These parameters have been extensively described in the literature [22]. As seen, the samples prepared at 250 W exhibited the highest roughness values (Ra and Rq) whereas the lowest values were obtained at 350 W. This observation at 350 W can be attributed to the fact that at high power, the energy of the particles increases with the sputtering power resulting in higher surface mobility and hence lower surface roughness [23]. Additionally, the lowest Rt and Rt values were obtained on surfaces deposited at 350 W. The highest values Rt and Rp were obtained at 150 W and 250 W respectively.

TABLE I. SURFACE PROFILE PARAMETERS

Power (W)	Surface Parameters			
	Ra (um)	Rq (um)	Rt (um)	Rp (um)
150	5.83±0.02	7.66±0.01	86.34±0.08	32.78±1.03
250	6.64±0.05	8.77±0.03	81.28±0.15	46.48±2.02
300	5.25±0.01	7.31±0.01	71.99±0.05	43.09±1.52
350	3.43±0.07	4.46±0.02	56.50±0.03	25.98±1.94

TABLE II. NANOINDENTATION PROPERTIES

Power (W)	Parameters		
	Contact depth (nm)	E (GPa)	H (GPa)
150	11.89±4.63	243.41±39.07	6.89±1.87
250	28.92±8.84	162.49±27.12	4.93±2.07
300	54.43±75.13	169.98±74.48	4.90±3.42
350	20.47±117.06	191.14±80.01	5.85±3.82

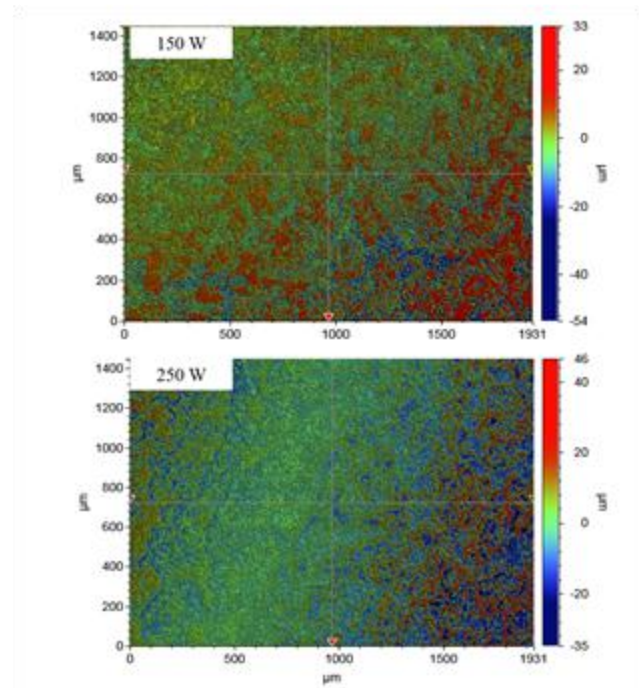


Figure 1. Optical surface profiling images for Al films sputtered at 150 W and 250 W.

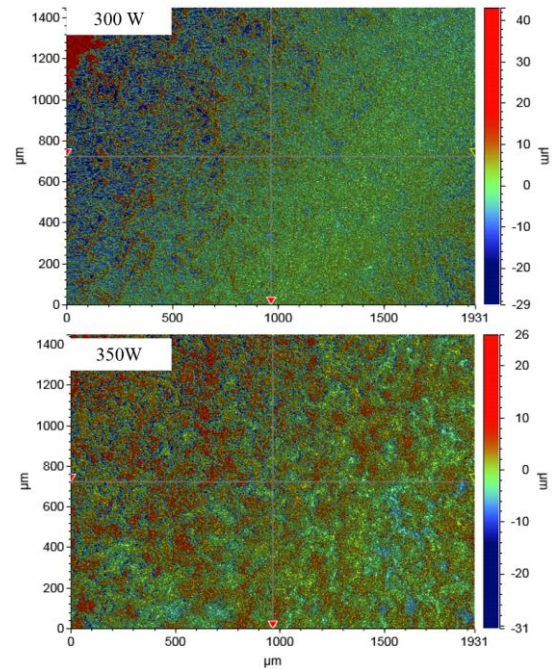


Figure 2. Optical surface profiling images for Al films sputtered at 300 W and 250 W.

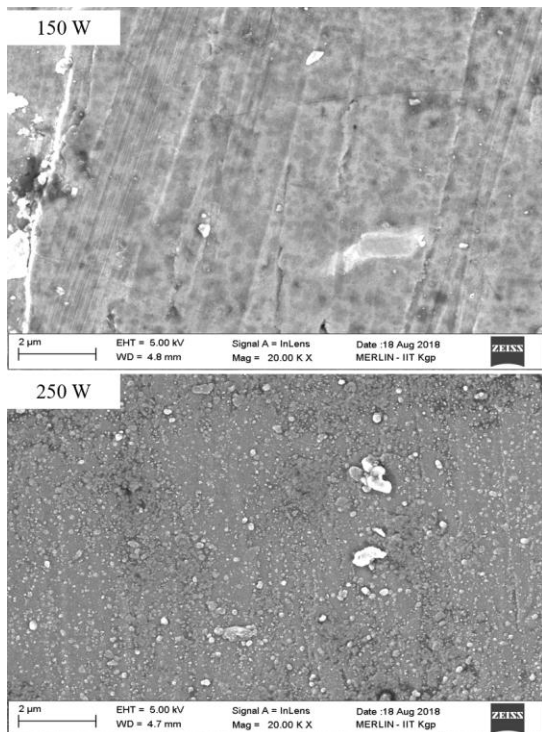


Figure 3. FESEM micrographs of Al thin films sputtered at 150 W and 250 W on mild steel substrates.

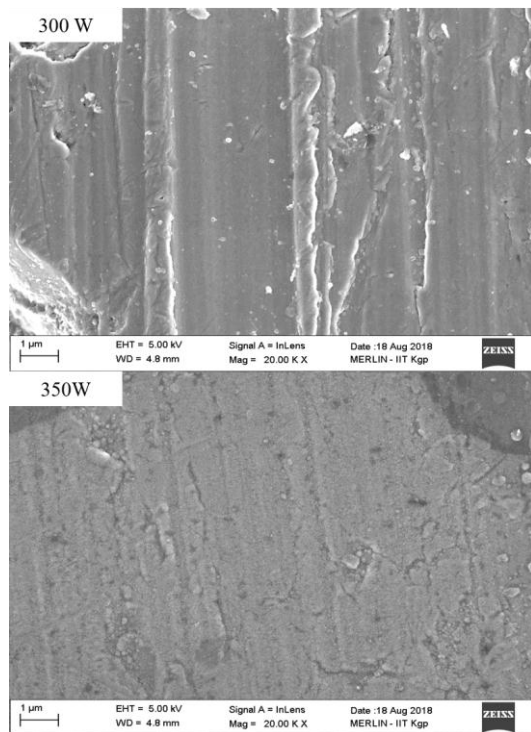


Figure 4. FESEM micrographs of Al thin films sputtered at RF power of 300 W and 350 W on mild steel substrates.

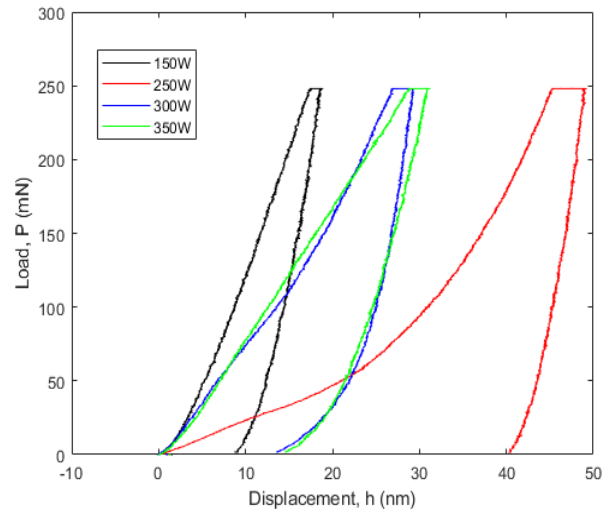


Figure 5. Nanoindentation curves (P-h) for the Al thin films sputtered at different RF powers.

Figs. 3 and 4 show the FESEM micrographs obtained from the surface of the aluminum thin films deposited at different RF powers. Amorphous microstructures exhibiting rough, porous and oxide (dark regions) structures with very few and fine Al structures were observed at 150 W. The very high R_t values obtained at 150 W can be attributed to porosity, oxides and amorphous structures of Al. At 250 W, visible particulates of Al were observed within the amorphous structure. There were also larger globular Al morphologies present at 250 W, which were interpreted as microstructural hillocks. The high surface roughness obtained at 250 W can be attributed to the presence of the hillocks. Larger and long structures of Al thin films were observed at 300 and 350 W. However, at 350 W, the structures appeared well-defined and relatively circular; clear secondary grains of Al films were observed. These microstructural observations suggest that the films deposited at 350 W were more crystalline as compared to those deposited at lower RF powers. Similar results on crystallinity have been reported for TaN thin films deposited via magnetron sputtering at varying RF powers in the range of 100-300 W [23].

Fig. 5 shows the load-displacement (P-h) curves for all the samples from the nanoindentation tests conducted using load control scheme. Both the loading and unloading curves indicated the occurrence of elastic-plastic behavior during the nanoindentation deformation. The contact depth, Young's modulus (E) and hardness (H) were derived from these curves based on the existing methodology [24], [25]. These values are summarized in Table II.

As seen in Table II, the highest values of H, E, and lowest contact depth were obtained at 150 W. This observation can be attributed two-fold reasoning: (i) FESEM images revealed the existence of oxide structures on the surfaces of the samples. The hardness and modulus values of such are oxides are generally higher than the Al films and (ii) at 150 W (low RF power), the sputtering yield could have been too low for full coverage of the mild steel substrate such that the values

obtained by the nanoindentation tests reflected those of the mild steel. Starting from 250 W to 350 W, the values of H and E increased proportionally with the sputtering power. At 350 W, the highest H and E values can be attributed to the formation of highly crystalline Al thin films.

The observations presented here indicate, indeed, a significant influence of the RF power to the properties of the Al thin films. The evolution can be explained by the implications of the power to the sputtering process. It has been shown that an increase in RF power increases the deposition rate. At high power, there is a high density of sputtered target atoms and more energetic plasma ions, which then impinge on the substrate surface creating defects on their respective points of impact. These defective areas exhibit higher adhesion energy for nucleation and hence accelerate the rate of growth of the film. However, it should be noted that at some very high value of energy (based on target and other processing conditions), argon (Ar) and the high energy particles can erode the substrate leading to very high roughness and high density of defects. As such, there exists an optimal sputtering power for each deposition process. In this case, the films deposited at 350 W exhibited attractive topography, microstructural and mechanical properties as compared to the other powers.

iv. Conclusions

The effect of power on the microstructure, roughness and nanoindentation properties of aluminum thin films prepared via RF magnetron sputtering was reported in this article. The following conclusions can be drawn:

- ❖ The surface roughness increased from 150 W to a maximum value at 250 W and then decreased steadily to a minimum value at 350 W. The behavior can be attributed to the effect of high energy atoms ejected and deposited onto the substrates.
- ❖ The microstructure of the Al thin films at low power (150 W) exhibited amorphous structure whereas at 350 W crystalline structures were observed through the FESEM. It implies that the crystallinity of the Al thin films increases with the sputtering power.
- ❖ The maximum Young's modulus and hardness values were obtained at 150 W and then decreased to a minimum at 250 W. The values increased from 250 W to 350 W. Therefore, the strength properties of Al thin films increase with the increase in sputtering power for porosity and oxide-free structures.

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