Time Influence on Thickness and Grains for Molybdenum Thin Film

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***Abstract*—In this paper, DC magnetron sputtering technique was used to deposit high purity molybdenum (Mo) thin films on blank Si substrate. The deposition condition for all samples has not been changed except for the deposition time in order to study the time influence on the surface morphology of the molybdenum. The surface profiler has been sued to measure the surface thickness. Atomic force microscopy technique was employed to investigate grain structure of Mo thin film. Grains analysis and thickness for all samples show a direct relation with time. The thickness and grain parameters of molybdenum thin films increase with respect to time. Grain area, size, length and perimeter parameters are used in grain analysis.**

***Index Terms*—Atomic Force Microscopy; DC Magnetron Sputtering; Surface Profile; Thin Film.**

I. Introduction

In recent years microelectromechanical systems (MEMS) have been developed following the improvement of nanotechnology. Precisely the development of lithographic processes enables the fabrication of a wide variety of material-based miniaturized devices [1- 4]. These systems have an increasing importance in the automotive industry, in magnetic storage devices and in all those applications where micro sensors or micro actuators are necessary. Thus it is crucial to face the new problems related to reduce dimensionality, in particular all the effects concerning the life cycle. MEMS are 10–100 times smaller than macro-machines, therefore surface forces often exceeds the volume forces and problems associated with friction/adhesion, wear and surface contamination become relevant. In this context, tribological studies have a key role in the optimization of these components [5-6].

Thin films and coatings play a critical role in everything from food containers to photovoltaic [7-9]. To meet such varied needs, they are made from every class of material and by numerous processes including physical and chemical vapor deposition techniques, atomic layer deposition, and sol gel processing [10]. A key step in developing any new film is characterizing its surface structure and physical properties, whether in engineering commercial products [11] or pursuing fundamental materials science [12].

Molybdenum (Mo) is a promising material to be used as electrodes in microelectronics and the deposited thin film can possess the interesting properties of molybdenum such as electrical conductivity [13] and its chemical stability. Molybdenum is commonly used as electrodes because of its ohmic contact to silicon [14], therefore characterizing molybdenum thin film toward utilizing it in microelectronic is very important in order to be used in some application. At present the sputtering deposition technique is widely used among the researcher to fabricate thin film because its advantages such as smaller grain size, many grain orientation and better adhesion [15], smaller grain impedes the dislocation motion and improve toughness as will.

In this work molybdenum thin film is deposited and characterized. DC magnetron sputtering which is a method of physical vapor deposition of thin film is used; it is considered one of the most common used and one of the fastest growing processes [16]. One of scanning probe microscopy (SPM) mode which is Atomic Force Microscopy (AFM) technique is used to characterize the samples [17-18]. Grains was studied for the samples and analyzed by the image analyses software (IA-P9). While the thickness is measured with a surface profiler.

II. Material and Methods

A. *Sputtering Process (Film Deposition)*

To prepare molybdenum thin film (99.95%) pure molybdenum round target is used with 5 inch diameter and with 0.25 inch thickness deposited on blank (1×1 inch2) Si substrate by DC magnetron sputtering process, the substrates were not subjected to any heating treatment before the sputtering process. Table 1 shows the input parameters for the DC magnetron.

The desire to have a specified thickness makes us change the time parameter only and leave other parameter constant; there will be 5 samples according to time changes.

Table 1
Input Parameter and It's Range

Input parameter	Range
DC power	200 Watt
Working pressure	10 mTorr
Argon flow	20 sccm
Deposition Time	(5 – 85) minute

We used the magnetron sputtering system and we can describe the machine by the following specifications:

-500x500 mm vacuum chamber
-3 selectable power supply (DC, MF, and RF)
-2-5 magnetron source with shutters
-3 selectable substrate temperatures (400, 600, 800 c)
-Rotating substrate holder with water cooling
-1600, 2000 L/s vacuum pump selectable
-Pressure control by capacitance manometer
-PLC system controlling, safety interlock, industrial PC and touch screen
-1.7(L) ×1.2(W) ×2.0(H) m system dimension

B. Morphological Characterization Using AFM

For our samples, morphology and surface texture have been studied using AFM technique. AFM as an excellent device is one of the most common techniques which are widely used in thin film characterization. Knowing the surface topography at Nano metric resolution made it possible to investigate dynamic biological process [19], tribological properties [20], mechanical manufacturing [21] and mainly thin film surfaces [22].

Researchers use AFM technique because it allows evaluation and precise observation of thin film characteristics. Which make it more preferable compare to other microscopic techniques? Furthermore, AFM can operate in ambient condition and does not need any special sample preparation [23]. After we obtained 2D and 3D images by AFM technique in non-contact mode, the resulted images were analyzed using prober software called Image Analysis-P9, which can gave a good and meticulous observation characterization parameters. In addition, IA-P9 software can process and analyze images and data from AFM, it also contains many techniques 2D and 3D function of data such as surface roughness, and grain analysis [24]. Figures, tables, charts & diagram should be kept to a minimum. They must be in black and white with minimum shading and numbered consecutively. They should have a brief title/caption in a font size of 8.

III. Results and Discussion

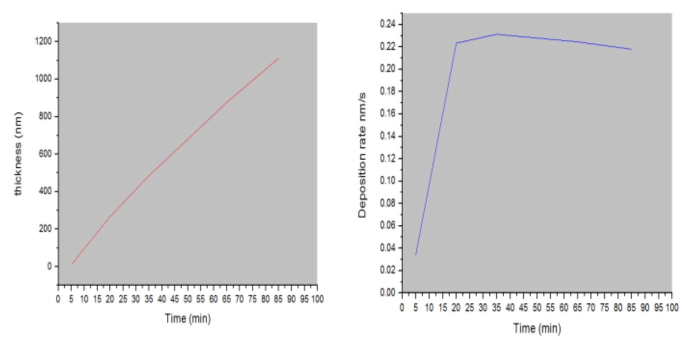
A. Thin Film Thickness

The measurement accuracy of thin film thickness is very important for many applications like semiconductor devices, displays, and thin film for optical product coatings. Average thickness can be determined by knowing the average step height (ASH) at any location in the scan area using surface profiler dektak150. The result for our sample was done by using tape all along Si substrate before the deposition process, this tape removed after deposition, thus there will be an area don't have the molybdenum, there are 4 test done in every sample with deferent places with area maximum equal (100×100) µm2. Table 2 shows the result and the average four all samples and the deposition rate which calculated by divide the average thickness on time.

The results show that the deposition rate became stable for all samples after 20 minute and for 5 minute it very small, at the beginning of deposition process the deposition rate will be small and start to increase with time to the point that became constant. Figure 1(a, and b) present the trends between time and thickness, and the trend between time and deposition rate respectively.

Table 2
Thickness and The Average Thickness for Mo Thin film With Different Deposition Time

Unit	nm					nm/s
deposition time (min)	Test 1	Test 2	Test 3	Test 4	Average	Deposition rate
5	9.27	10.9	12.4	7.89	10.115	0.0337
20	269.96	266.75	268.28	267.44	268.107	0.2234
35	498.76	491.67	472.11	489.06	485.65	0.2312
65	876.18	878.90	872.44	876.45	875.992	0.2246
85	1114.4	1116.3	1079.4	1137.2	1111.87	0.2180



a b

Figure 1: Correlation between time and a) thickness, and b) deposition rate.

B. Grain Analysis

Grain Analysis method provides visualizing the section of the grain ensemble taken at a predefined relative level common for all grains, it collecting basic geometric characteristics of particles in the ensemble, including section area, volume, average size, local height, maximum height, maximum size, average height, perimeter, and collecting the distribution of a particular geometric characteristic and visualizing it as a histogram. Grain Analysis method is applicable to analysis AFM images of granular ensembles on the surface under few assumptions includes the particles of the ensemble are located on a base surface, shape of the particles is sufficiently convex, and the particles are separated.

In previous images for Mo samples, IA-P9 image processing software analyzed and generated quantitative information for both individual and group of grains. On a group of particles, a statistical measurement can be gathered. Furthermore, counts of particles and distribution of all size of particles, surface area, and volume are the most common statistic measurement. However, for individual grain, physical

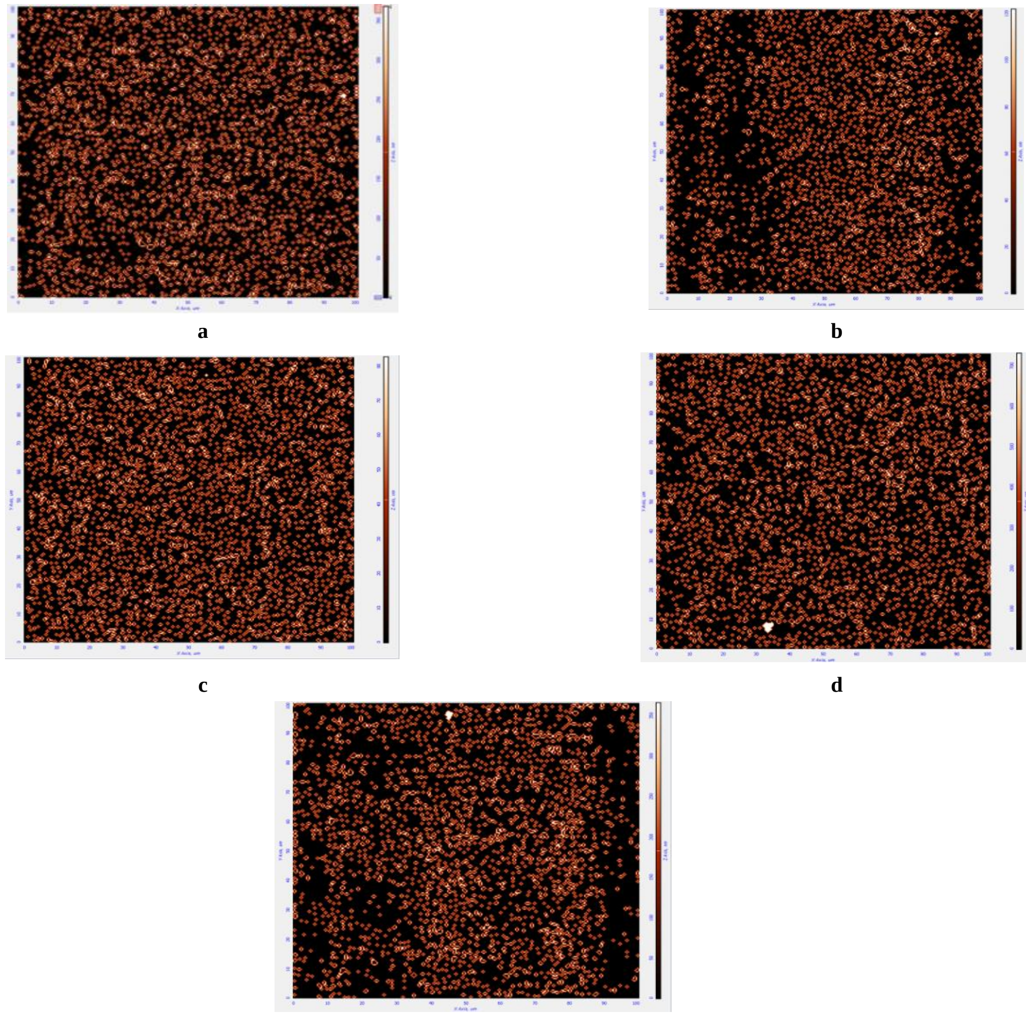


Figure 2: 2D image show the grain distribution of Mo films with deferent deposition time, a) 5min b) 20 min c) 35min d) 65min e) 85min.

properties such as surface texture, morphology, and 3D size information like (height, length, and width) can be measured using same software. 2D image shows grain distribution for Mo thin film with different deposition time from IA-P9 shown in Figure 2, the images shows the good distribution of the grain all over the area.

Statistical information is shown in Table 3 includes the distribution of all measured parameters (grain area, grain size, length and perimeter) of Mo thin films nanostructure with different deposition time. Grain size, area, perimeter, and length increase with respect to time. Small grain size is detected, which is preferable. As mentioned before small grain size increases the films toughness.

Figure 3 illustrate histogram plots of Quantitative analysis for Mo thin films with different deposition time, the images show a very good grain distribution all over the area.

Table 3
Grain Analysis of Mo Thin Film according to Deposition Time

Unit	minute	Average			
		µm^2	µm		
Sample number	deposition time	Area	size	Perimeter	Length
1	5 min	0.0035	0.046	0.19	0.068
2	20 min	0.017	0.12	0.48	0.175
3	35 min	0.0011	0.03	0.12	0.04
4	65 min	0.0023	0.04	0.175	0.058
5	85 min	0.003	0.045	0.2	0.065

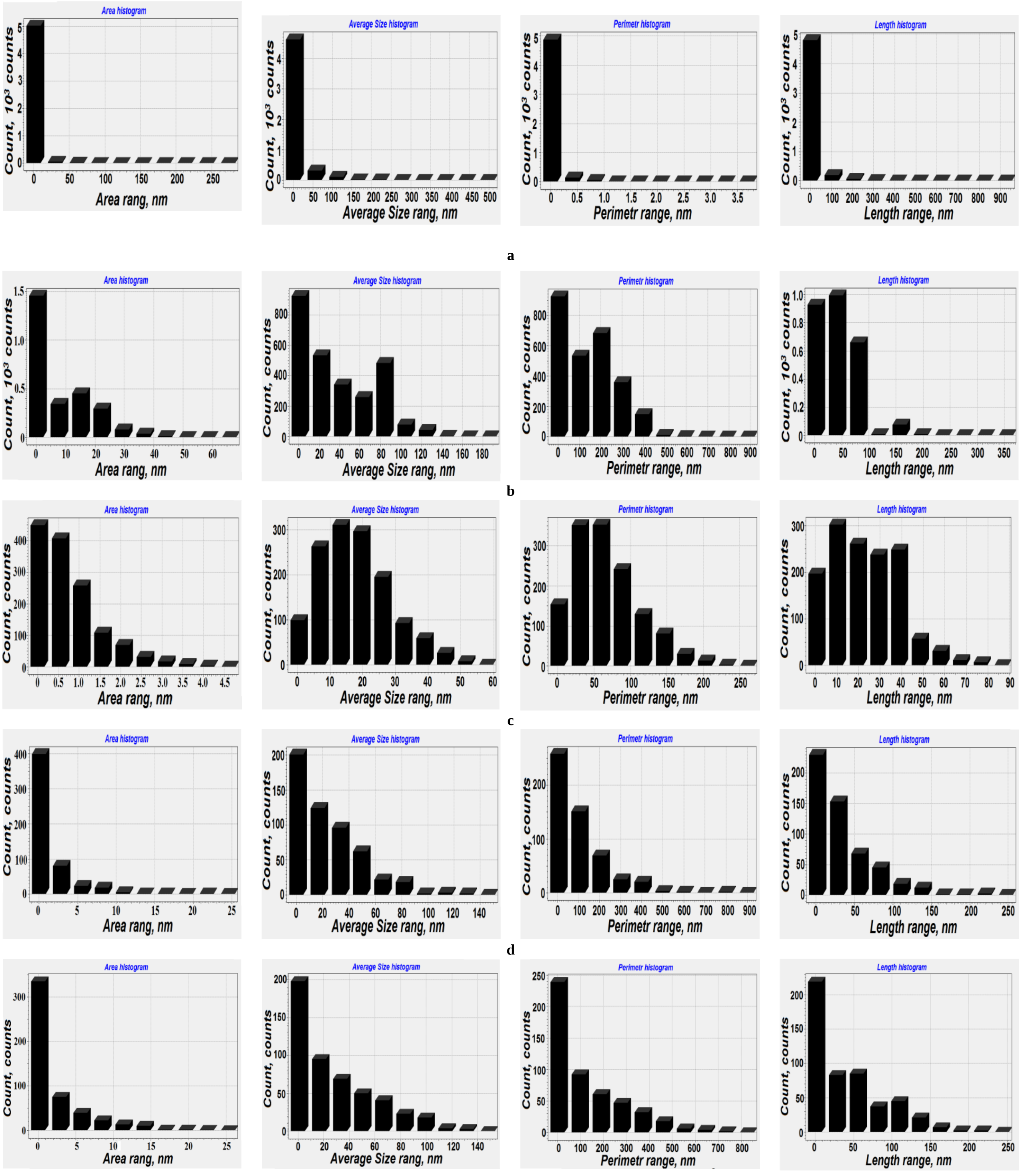


Figure 5: Quantitative analysis of nanostructured for Mo thin film with different deposition time, a) 5min b) 20 min c) 35min d) 65min e) 85min.

IV. CONCLUSION

In this work, AFM used to characterize surface thickness and grain analysis of Mo thin film deposited on Si substrate with deferent deposition time rang (5 – 85) minute. Image analysis P9 used to process the data from AFM and get the statistic such as 2D, 3D, and histogram. Deposition rate for all samples were calculated, it equal 0.0337 nm/s for the 5 minute sample and it increase for other sample to the point that remain constant after 20 min to be 0.22 nm. The grain analysis for all sample showed that grain parameter value increase with respect to time, and very good distribution of the grain along the surface. This type of study provides more extensive understanding of the influence of time on surface morphology of the films and could help in choosing the deposition parameters according to surface morphology requirements for any application.

ACKNOWLEDGMENT

This work has been supported by Ministry of Science, Technology & Innovation (MOSTI) under Grant (03-01-14-SF0095). The authors would like to acknowledge the Center of Research and Innovation Management (CRIM) in Universiti Teknikal Malaysia Melaka (UTeM) and Universiti Kebangsaan Malaysia (UKM).

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