

Research Article

Deposition and Characterization of Molybdenum Thin Film Using Direct Current Magnetron and Atomic Force Microscopy

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In this paper, pure molybdenum (Mo) thin film has been deposited on blank Si substrate by DC magnetron sputtering technique. The deposition condition for all samples has not been changed except for the deposition time in order to study the influence of time on the thickness and surface morphology of molybdenum thin film. The surface profiler has been used to measure the surface thickness. Atomic force microscopy technique was employed to investigate the roughness and grain structure of Mo thin film. The thickness and grain of molybdenum thin film layer has been found to increase with respect to time, while the surface roughness decreases. The average roughness, root mean square roughness, surface skewness, and surface kurtosis parameters are used to analyze the surface morphology of Mo thin film. Smooth surface has been observed. From grain analysis, a uniform grain distribution along the surface has been found. The obtained results allowed us to decide the optimal time to deposit molybdenum thin film layer of 20–100 nm thickness and subsequently patterned as electrodes (source/drain) in carbon nanotube-channel transistor.

1. Introduction

In recent years, the researches in microelectromechanical systems (MEMS) have developed remarkably, following the advanced of nanotechnology. The development of lithographic processes enables the fabrication of a wide variety of material-based miniaturized devices [1–4]. These systems have a rising importance in the automotive industry, magnetic storage devices, and all of those applications where microsensors or microactuators are necessary. Thus, it is crucial to face the newly emerged problems related to the reduce dimensionality. MEMS are 10–100 times smaller than macromachines; therefore surface forces often exceed 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 studies [12].

Molybdenum (Mo) is a promising material to be used as electrodes (source/drain) in microelectronics. Mo thin film possesses interesting properties such as high electrical conductivity [13] and good chemical stability. Molybdenum is

TABLE 1: Input parameters for sputtering.

Input parameter	Range
DC power	200 Watt
Working pressure	10 mTorr
Argon flow	20 sccm
Deposition time	5-85 minutes

commonly used as electrodes because of its ohmic contact to silicon [14]. Therefore, molybdenum thin film characterization towards microelectronic utilization is very important for some applications such as in carbon nanotube transistor and resonant gate transistor [15, 16]. The sputtering deposition technique is widely used among the researchers to fabricate thin film due to its advantages such as creating thin films with smaller grain size, many grain orientation, and better adhesion with the substrate [17]. Smaller grains impede the dislocation motion and improve toughness as well.

Recently, many researches have been done on the deposition of molybdenum thin films, electrical and morphological studies of Mo thin film for solar cell, and mechanical and tribological studies of molybdenum nitride thin films [13, 18– 22]. Nevertheless, no study has been carried out on electrodes (source/drain) fabrication which needs specific thickness and grain structure in nanodevices, for example, in the growth of carbon nanotube as channel between the electrodes (source/drain).

In this work, molybdenum thin film is deposited and characterized in order to be used as electrodes for carbon nanotube transistor. Thin film molybdenum layer has been deposited on a silicon wafer. DC magnetron sputtering which is a method of physical vapor deposition technique for thin film is used; it is considered to be one of the most commonly used techniques [23]. In this study, the influence of deposition time on thickness, grain, and roughness of Mo thin film layer has been carried out while argon flow rate, DC power, and working pressure have been made constant during deposition. One of scanning probe microscopy (SPM) mode which is atomic force microscopy (AFM) technique is used to characterize the samples [24, 25]. Surface roughness and grain analysis of the samples are analyzed by Image Analyses-P9 (IA-P9) while the thickness is measured with a surface profiler.

2. Material and Methods

2.1. Sputtering Process (Film Deposition). In DC magnetron sputtering process, pure molybdenum round target (99.95%) with 5 inch diameter and 0.25 inch thickness has been used to deposit Mo on blank (1×1 inch²) Si substrate. The substrates have not been subjected to any heating treatment before the sputtering process. Table 1 shows the input parameters for the sputtering process.

There are 5 samples of Mo thin film layer deposited at different sputtering time from 5 minutes to 85 minutes. Specific Mo thickness for carbon nanotube transistor's electrode can be achieved by varying the sputtering time. 2.2. Morphological Characteristic 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 nanometric resolution allows researchers to investigate dynamic biological process [26], tribological properties [27], mechanical manufacturing [28], and mainly thin film surfaces [29].

Researchers use AFM technique because it allows evaluation and precise observation of thin film characteristics. Furthermore, AFM can operate in ambient condition and does not need any special sample preparation [30]. The most commonly used parameters to study surface texture include roughness, waviness, flows, and lay. All of these parameters represent random deviation of the surface. After we obtained 2D and 3D images by AFM technique in noncontact mode, the resulting images were analyzed in AP-9 software.

2.3. Surface Roughness Analysis. The most common parameters measured for roughness were the roughness average (R_a) and the root mean square (R_q) , sometime called RMS. R_a is the arithmetic average value of the deviation of the trace above and below the mean value μ (center line). In other words, R_a is a vertical deviation which is the mean heights variation of the surface area according to the reference plan [31]. RMS roughness (R_q) measures the root mean square deviation of a profile and is used in calculating the skew and kurtosis parameter. R_q values found for all sample were higher than R_a . The mathematical explanation of R_a and R_q is given by the following equations:

$$R_{\rm a} = \frac{1}{Nx * Ny} \sum_{j=1}^{Ny} \sum_{i=1}^{Nx} |Zij - \mu|, \qquad (1)$$

where Nx * Ny is data sample size in the array Zij which is the source discrete function in the *XY* plane and μ is the mean value the first momentum of the distribution given by

$$\mu = \frac{1}{Nx * Ny} \sum_{j=1}^{Ny} \sum_{i=1}^{Nx} Z_{ij}$$
(2)

$$R_{\rm q} = \sqrt{\frac{1}{Nx * Ny} \sum_{j=1}^{Ny} \sum_{i=1}^{Nx} (Zij - \mu)^2}.$$
 (3)

Surface skewness (R_{sk}) characterizes the symmetry of distribution. It is nonzero for symmetric distributions, positive for distributions with dominating right tail, and negative for distributions with dominating left tail. If the value of R_{sk} is positive, peaks become dominant in the distribution and if it is negative, valleys become dominant in the distribution [32]. Surface skewness is given by

$$R_{\rm sk} = \frac{1}{Nx * NyR_{\rm q}^3} \sum_{j=1}^{Ny} \sum_{i=1}^{Nx} \left(Zij - \mu \right)^3. \tag{4}$$

Coefficient of kurtosis (R_{ka}) is a measurement of spikiness distribution profile, above and below the reference plan. The

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	268.1075	0.2234
35	498.76	491.67	472.11	489.06	485.65	0.2312
65	876.18	878.90	872.44	876.45	875.9925	0.2246
85	1114.40	1116.37	1079.44	1137.29	1111.875	0.2180



FIGURE 1: Trend between (a) thickness and (b) deposition rate with respect to time.

surface is considered Gaussian if $R_{ka} = 3$, spiky if $R_{ka} > 3$, or bumpy for $R_{ka} < 3$ [32]. The following formula presents R_{ka} :

$$R_{\rm ka} = \frac{1}{Nx * Ny R_{\rm q}^4} \sum_{j=1}^{Ny} \sum_{i=1}^{Nx} \left(Zij - \mu \right)^4 - 3. \tag{5}$$

Skewness and kurtosis parameter are important in giving better understanding of the surface. Skewness parameter indicates whether the peak distribution is symmetrical while kurtosis parameter determines whether the distribution of height (histogram) is Gaussian.

3. Results and Discussion

3.1. 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. Four measurements have been done in every sample at different places. Table 2 shows the thickness measurement and deposition rate. Figures 1(a) and 1(b) present the trends between time and thickness and the trend between time and deposition rate, respectively. In Figure 1(a), the thickness of Mo thin film layer increases linearly with respect to time. The result in Figure 1(b) shows that the deposition rate at the beginning of deposition process is small and starts to increase with time to the point that it became constant. The deposition rate has been measured to be constant for all samples deposited for more than 20 minutes.

3.2. Surface Roughness. Like thickness, surface roughness analysis is important to thin film due to its contribution in both mechanical and electrical transport properties. Conducting thin film roughness has a tangible impact on device performance [33]. 2D and 3D AFM images for Mo film are shown in Figure 2 for time deposition at 5 minutes to 85 minutes.

In Figure 3, the histogram and peak distribution are presented. Histogram is the heights distribution and it possesses a bell shape. Peak is the accumulated heights distribution. In order to clearly explain Figure 3, we should understand the statistical value parameter and amplitude which help to clarify the histogram shape and peak distribution. It is necessary to know the amplitudes for R_a and R_q calculated by (1) and (2), respectively. High R_a means rough surface; small R_a means smooth surface. Smooth surface is usually more resistive than rough surface against friction and wear. Our samples have low roughness as shown in Table 3. According to [34], the height distribution of most surfaces may approach a Gaussian distribution if R_q/R_a value is up



FIGURE 2: Continued.



FIGURE 2: 2D and 3D AFM image samples with different deposition time, (a) 5 min, (b) 20 min, (c) 35 min, (d) 65 min, and (e) 85 min.

TABLE 3: Roughness parameter of Mo thin film according to different deposition time.

Unit	n	m	R/R	R	R
Deposition time (min)	$R_{\rm a}$	R_{q}	n_q/n_a	R _{sk}	T _{ka}
5	1.33	1.69	1.27	0.22	3.7
20	2.58	3.23	1.25	0.35	2.97
35	2.44	3.06	1.25	0.33	3.04
65	1.57	1.99	1.26	0.21	4.7
85	1.17	1.46	1.25	0.22	2.9

to 1.31. The calculated values for $R_{\rm q}/R_{\rm a}$ in our experiment for all samples are approximately equal to 1.25 which means that for all 5 samples, the height distributions tend to be Gaussian. Referring to skewness and kurtosis definition in Section 3.2, these parameters describe the height symmetry. In our experiment, the values for skewness for all samples, which has been calculated using (4), are positive in a range of 0.21-0.35. Thus, the peak distribution in Figure 3 shows that the right tail is longer than left tail. In addition, the hills are dominant over the valleys which indicate that the distributions are not perfectly symmetric. Values for Kurtosis as calculated in (5) are greater than 3 for samples with deposition time of 5 minutes, 35 minutes, and 65 minutes which indicate that the surface is spiky and the distribution is leptokurtic [23]. However, the surface is bumpy and the distribution is platykurtic [23] for samples with 20-minute and 85-minute deposition time, related to kurtosis value of less than 3.

The results clearly show that the roughness of molybdenum thin films decreases with respect to time. Furthermore, the films surfaces have waviness surface texture. For all samples, the histogram distributions are Gaussian and the peak distributions are dominant over the valleys.

3.3. *Grain Analysis*. Grain analysis method visualizes the section of the grain ensemble taken at a predefined relative level common for all grains. It collects basic geometric

TABLE 4: Grain analysis of Mo thin film according to deposition time.

		Average				
Unit	μm^2	$\mu { m m}$				
Deposition time (min)	Area	Size	Perimeter	Length		
5	0.0035	0.046	0.19	0.068		
20	0.017	0.12	0.48	0.175		
35	0.0011	0.03	0.12	0.04		
65	0.0023	0.04	0.175	0.058		
85	0.003	0.045	0.2	0.065		

characteristics of particles in the ensemble, including section area, volume, average size, local height, maximum height, maximum size, average height, and perimeter. A particular geometric characteristic for a section of grain ensembles are collected and presented in a histogram. Grain analysis method analyzes AFM images of granular ensembles on the surface under few assumptions, including that the particles of the ensemble are located on a base surface, the 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. In a group of particles, a statistical measurement can be gathered. Furthermore, counts of particles and distribution of all particle sizes, surface area, and volume are the most common statistic measurement. For individual grain, physical properties such as surface texture, morphology, and 3D size information (height, length, and width) can be measured using the same software. In Figure 4, 2D images show grain distribution for Mo thin film with different deposition time. The images show very good distribution of grain on all over the sample area. Figure 5 illustrates 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 4 concludes the average of all measured parameters (grain



FIGURE 3: AFM roughness analysis with different deposition time, (a) 5 min, (b) 20 min, (c) 35 min, (d) 65 min, and (e) 85 min. First column for histogram and the second for peak distribution.



(e)

FIGURE 4: 2D image shows the grain distribution of Mo films with different deposition time, (a) $5 \min$, (b) $20 \min$, (c) $35 \min$, (d) $65 \min$, and (e) $85 \min$.



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

area, grain size, length, and perimeter) of Mo thin films nanostructure with different deposition time. Small grain size is preferable as it increases the films toughness.

4. Conclusions

In this work surface profiler and AFM have been used to characterize surface thickness, roughness, and grain analysis of Mo thin film deposited on Si substrate with different deposition time (5-85 minutes). Image analysis P9 has been used to process the data from AFM and produce the statistical information such as 2D, 3D, and histogram. Deposition rate for all samples has been calculated and it has been found to be 0.0337 nm/s for sample with 5-minute deposition time. The deposition rate increases for other samples to the point that it remains constant after 20 min at 0.22 nm/s. The result showed that the films surfaces have smooth surface texture. For all samples, the distributions are Gaussian and the peaks are dominant over the valleys. The surface roughness decreases with time. The grain analysis for all samples showed that the grain parameter values increase with respect to time and very good distribution of grain along the surface. This type of study provides more extensive understanding of the influence of time on thickness and surface morphology of the films. Other than the deposition time, similar analysis could also be made with variation of DC sputtering power, sputtering pressure, and sputtering argon flow rate. This could help in choosing suitable deposition parameters according to thickness and surface morphology requirements for any application such as fabrication of the electrode for the carbon nanotube transistor.

Competing Interests

The authors declare that they have no competing interests.

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