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ORIGINAL ARTICLE

Preparation and characterization of DC sputtered molybdenum thin films

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Abstract Molybdenum (Mo) thin films have been deposited on soda-lime glass substrates using a DC magnetron sputtering system. Their electrical resistivity, and their morphological, structural and adhesive properties have been examined with respect to the deposition power, deposition time and substrate temperature. The electrical resistivity of the Mo films could be reduced by increasing any of the above parameters. Within the range of the investigated deposition parameters, the films showed a mono-crystalline nature with a preferred orientation along the (1 1 0) plane. The Mo films adhesion to the soda-lime glass could be improved by increasing the substrate temperature. At a deposition power of 200 W, deposition time of 20 min and substrate temperature of 450 °C, Mo thin film exhibiting mono-crystalline structure with thickness equal to 450 nm and electrical resistivity equal to $1.85 \times 10^{-4}\ \Omega$ cm was obtained.

1. Introduction

Because of its low resistivity and high melting point, sputter-deposited thin films of molybdenum (Mo) are increasingly being used as the gate electrode in GaAs-based metal gate field effect transistors (MESFETs) and silicon-based metal–oxide-semiconductor (MOS) devices [2,4,5,9]. Successful performance of Mo as the gate electrode was found to be affected by the deposition conditions. Lin et al. [5] deposited pure Mo films on steam-oxidized Si wafers by DC magnetron sputtering in argon plasma. He reported that the resistivity of Mo films shows a pronounced decrease with an increase in the negative substrate bias and the rate of film growth. The changes in

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the resistivity of Mo films were attributed to the degree of oxygen contamination and microstructural modifications during growth. This explanation was sustained by Bardin et al. [2] who showed that the electrical resistivity of Mo films decreases as the amount of oxygen contamination decreases at higher deposition rates. Yamaghuchi and Miyagawa [9] showed also that the sputtered Mo thin films microstructures and stresses were affected by oxygen. The fibrous grain width decreases as oxygen content increases because the Mo atom migration is suppressed by the Mo surface oxidation during deposition. The bulk film stresses changed from tension to compression as the oxygen content in the films increased.

Mo also emerged as the dominant choice for back contact layer of $CuInSe_2$ (CIS) and its gallium alloys (CIGS)-based solar cells because of its relative stability at the processing temperature, resistance to alloying with Cu and In, and its low contact resistance to CIS [7,8,6,1,3]. As the quality of the CIS film, i.e. morphology, grain size, orientation, etc., was found to be directly affected by the quality of the underlying Mo layer. Martinez and Guillén [6] studied the optoelectronic, morphological and structural properties of r.f. magnetron-sputtered Mo layers as related to the different deposition conditions. They concluded that all samples have a comparable electrical behavior but low r.f. power densities are necessary to minimize stresses and obtain densely packed structures. Besides, low Argon mass-flow rates can reduce micropores and increase optical reflectance. Gordillo et al. [3] deposited Mo thin films using a DC magnetron sputtering system with S-gun configuration electrode. They showed that it is possible to get low resistivity Mo films by decreasing the Argon partial pressure and increasing the power of the glow discharge.

As the properties of the deposited Mo layer play a crucial role in the performance of the above applications, our objective in this work was to investigate the effects of deposition parameters on the morphology, crystal structure, resistivity and adhesion of the Mo films. The parameters considered were the deposition power, the deposition time and the substrate temperature. Conditions that lead to low resistivity, monocrystalline Mo thin films were established.

2. Experimental details

Mo thin films were produced on 2.5 cm × 7.5 cm × 1.115 mm soda-lime glass substrates by means of a commercial DC magnetron sputtering system (HUMMER 8.1) using Mo target foil (diameter 10.16 cm, thickness 0.64 cm, purity 99.95%). Substrates were cleaned using soap and distilled water. After scrubbing with soap, the substrates were rinsed in running distilled water. Next, they were etched by a 0.1 M HCl aqueous solution and then washed in distilled water and acetone using an ultrasonic bath for 15 min. They were subsequently introduced into the vacuum chamber and placed at a distance of 10 cm from the Mo target.

The vacuum chamber was initially evacuated to a base pressure of 6×10^{-4} Torr. Then pure Argon (99.999%) flow was introduced into the chamber to keep the pressure during the film deposition at a value around 7.5 mTorr. The power supply was then turned on. A series of eighteen Mo films were prepared under different conditions for the deposition power, the substrate temperature and the deposition time of the deposition process. The selected deposition parameters are summarized in Table 1.

Table 1 Summary of deposition parameters for the Mo prepared thin films.

Deposition power (W)	Substrate temperature (°C)	Deposition time (min)
70	Room temperature	10
		20
	150	10
		20
	450	10
		20
150	Room temperature	10
		20
	150	10
		20
	450	10
		20
200	Room temperature	10
		20
	150	10
		20
	450	10
		20

The crystalline structure of the films was investigated by X-ray diffractometer (Schimadzu-7000) using Cu-Kα radiation target. The surface morphology and homogeneity were examined with high-resolution field emission scanning electron microscope (Zeiss Ultra 55). The resistivity of the coatings was measured using a four point probes Van der Pauw technique (Sigmaeone) and the reported value is an average of three measurements taken for each sample. The adhesion test was performed using cross hatch tape adhesion test (hatch cutter) with Cross Cut Adhesion Tester (CC2000) and ASTM D3359 with 1 mm spacing (Proinex Instrument. s.r.o., http://www.proinexinstruments.com).

3. Results and discussions

3.1. Electrical and morphological properties

The Van der Pauw method has been used to measure the electrical resistivity of the deposited Mo thin films. In conformity with Gordillo et al. [3] results, the resistivity was found to decrease by increasing either the deposition power or the deposition time as shown in Fig. 1. For each sample, the three measured values are plotted and the solid curves join the average values corresponding to each deposition time. From the figure, it can be seen that the deposition time is less effective as the deposition power increases. This can be explained by the fact that the high deposition power enhances the rapid growth of a relatively thick film. Since the increase of the film thickness leads to the decrease of the resistivity of the Mo films [3], the effect of the deposition time is reduced as the deposition power increases. Fig. 2 shows that the resistivity decreases also by increasing the substrate temperature. The film prepared at a deposition power of 200 W, a substrate temperature of 450 °C and a deposition time of 20 min was found to have a resistivity equal to $1.85 \times 10^{-4}\ \Omega$ cm, corresponding to the lowest resistivity value for the Mo thin films prepared in this work. This value lies within the range of values