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The effect of microstructure of molybdenum target

Geng An[1, 2,] Jun Sun[1],
[1] State key laboratory of mechanical behavior for materials, Xi'an Jiaotong University, Xi'an, 710049, China
[2]Technical Center, Jinduicheng Molybdenum Co., Ltd., Xi'an 710077, China

Abstract: Molybdenum (Mo) thin films were sputtered from two kinds of Mo targets with different microstructures under the same sputtering process, and the effect of microstructure of Mo target on morphology, deposition rate and resistance of sputtered film were studied and discussed. The results show that morphological differences between Mo thin films sputtered by Mo targets with different microstructures are very small. The more uniform and finer the grain structures of Mo target, the better the uniformity on thickness and resistance of Mo sputtered film. Moreover, during sputtering process, when Mo target's grain size is finer and the surface area of grain boundary is higher, the thickness reduction of the target is more homogeneous and the sputtering film has faster deposition velocity. The difference in microstructure of the Mo target has not obvious influence on the grain orientation of sputtering film.
Keywords: Preparation, molybdenum target, sputtered film, microstructure, grain orientation

1. Introduction

Molybdenum (Mo) is a typical refractory metal with many special properties, such as high temperature strength, low thermal expansion coefficient and high Young's modulus [1, 2]. In recent years, many studies have shown that using Mo thin films to replace or as barrier layers of Ag, Cu and Al thin films can effectively improve the conductivity, anti-electro migration and thermal stability of thin film electrode. Therefore, Mo is considered a thin film material with great potential and has been utilized extensively in electrodes or barriers for numerous optoelectronic applications, such as $CuIn_{1-x}Ga_xSe_2$ (CIGS)-based solar cells or thin film transistor-liquid crystal displays (TFT-LCD) [3-5].

Mo thin films are usually deposited by sputtering methods, and Mo targets were produced for thin film sputtering. The purity, density and microstructure of Mo target affect the characteristics of thin films. Therefore, one of the important preconditions for obtaining excellent Mo thin films is to produce a high-quality Mo target. Many studies focused on the sputtering process and evolution of thin film structures upon properties of molybdenum film [6-10], and fewer studies compared the effects of microstructures in Mo targets upon the properties of the thin film subsequently sputtered. The aim of this study was to gain understandings by looking into the microstructures of Mo targets. In this study, two kinds of Mo targets with different microstructures were made to sputter thin films. The effect of microstructure of Mo target on morphology, deposition rate and resistance of sputtered film were discussed.

2. Experimental

Two kinds of Mo targets with different microstructures were made to sputter thin films under the same sputtering process. They were marked as TXM and TYM, respectively, in this paper. Fig. 1 shows the microstructure of the two Mo targets. The grains of TXM Mo target are fine and uniform (Fig. 1a), and the average grain size is 36.2 μm. In contrast, the grains of TYM Mo target are non-uniform (Fig. 1b), and the average grain size is 86.7μm, which is also bigger than that of TYM Mo target. The purities and relative densities of both targets are all more than 99.95% and 98%.

The ULVAC ACS-4000-C4 magnetron sputtering equipment was used to carry out sputtering tests with both Mo targets in single target sputtering mode. Substrates were polished glass slides of 1.2 mm in thickness. They were ultrasonically washed with acetone and alcohol respectively for 20 min., as well as washed with deionized water for 10 min. before coating. Afterwards, the substrates were dried with nitrogen and placed into vacuum chambers. The initial vacuum degree of the sputtering system was 4×10^{-3} Pa. The vacuum degree was maintained at 0.133 Pa during the sputtering process of Mo thin films. Moreover, the sputtering was conducted with 99.999% high-purity argon flowing at 15 SCCM as sputtering gas at room temperature and the sputtering currents were adjusted to 3.0 A. After preparing thin films, the JSM-6700 field-emission scanning electron microscope (FESEM) was used to observe grain structure and morphology of Mo films. By utilizing the atomic force microscope (AFM), the surface roughness of the Mo films was observed. Furthermore, the thickness of Mo films was measured by the Dektak-XT stylus surface profilometer and four point probe technique was used to measure the resistivity of the Mo thin films. The reported grain size, thickness and resistivity data are an average of at least five measurements. The structure of Mo films was monitored by X-ray diffraction (XRD) using a PANalytical X pert PRO diffractometer with Cu Kα radiation.

3. Results

Fig. 2 exhibits the surface morphology of Mo thin films sputtered from two different targets. Fig. 3 shows

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the AFM images of two Mo films. It can been seen that morphological differences among two kinds of Mo thin film sputtered by Mo targets with different microstructures are very small, and Mo film are all made of “vermiform” grains. However, Mo thin film from TXM Mo target (marked as FXM) presented flat surface, and sputtering grains were chiselled and uniform in size. For Mo thin film from TYM Mo target (marked as FYM), its sputtering surface was relatively uneven (Fig. 2c and Fig. 3b) and sputtering grains were smooth and non-uniform in size (Fig. 2d). The surface roughness of FXM Mo film is 6.861 nm, which is smaller than that of FYM Mo film whose surface roughness is 9.047 nm.

The change of film thickness with sputtering time using two kinds of Mo targets is shown in Fig. 4. The TXM Mo target and TYM Mo target have deposition rates of 0.504 nm/ s and 0.467 nm/ s, respectively.

Fig. 5 shows the sheet resistance homogeneity of two kinds of Mo-target sputtered films. It is clear that sheet resistance homogeneity of FXM Mo film is better than that of FYM Mo film, and the average sheet resistance value of FXM Mo film is 326.14 mOhm/ sq, which is 17.1% lower than that of FXM Mo film, 393.43 mOhm/ sq.

Fig. 6 shows the XRD diffraction spectra of two Mo thin films. It can be seen that both thin films show good crystal quality and high single orientation. It is detected one single diffraction peak at 40.5°, corresponding to the grain orientation (110) of Mo. Therefore, the microstructural differences of Mo target have not obvious influence on grain orientation of the sputtering films.

Fig. 7 exhibits the surface and cross section morphology of two sputtered Mo targets. The surface dent of sputtered TXM Mo-target is fine and shallow (Fig. 7a), and the sputtered surface is relatively flat (Fig. 7b). However, sputtered TYM Mo-target presents a long and deep surface dent (Fig. 7c), whereas the sputtered surface displays deep sags and crests (Fig. 7d).

4. Discussions

The microstructure of TXM Mo target is mainly formed by fine and uniform equiaxed grains, but that of TYM Mo target presents a coarse grain structure. Therefore, the grain boundary area per unit volume of TXM Mo target is higher than that of TYM Mo target. Because atomic arrangement at the grain boundary is looser than the intracrystalline one, grain boundary is corroded more easily by mechanisms such as heat erosion or chemical corrosion. So, atoms at the grain boundary are relatively easy to be preferentially ejected when the target is ion bombarded at a given sputtering energy. The arrangement orientation of atoms in two grains is difference. Each grain strives to make the arrangement orientationof atoms tends to its own orientation. When the equilibrium state is reached, a certain transitional arrangement of atoms will be formed between the two sides of a grain boundary. Then, ion bombardment will expand from the grain boundary to the intracrystalline region. The bigger the grain boundary area per unit volume, the faster the sputtering along grain boundary and the expanding from the grain boundary to the intracrystalline region. In the case of TXM Mo target, there is no obvious orientation among grains and it has a homogeneous microstructure, which leads to the uniform ion bombardment of the target surface during sputtering process, and eventually, causes the fast sputtering of the whole target surface. Therefore, at the same sputtering time, the thickness of FXM Mo film is thicker than that of FYM Mo film (Fig. 4). On the other hand, the homogeneous microstructure of TXM Mo target leads to an uniform and flat surface of the film (Fig. 2a and Fig. 3a), and finally, causes a more homogeneous sheet resistance (Fig. 5).Therefore, the more uniform and finer the grain structure of Mo target, the better the uniformity of thickness and resistance of Mo sputtered film. Moreover, when Mo target's grain size is finer and the grain boundary area per unit volume is bigger, the thickness reduction of the target is more homogeneous and the sputtering film deposition is faster.

5. Conclusion

1) The morphological differences between Mo thin films sputtered from Mo targets with different microstructures are very small.
2) During the sputtering process, the more uniform and finer the grain structure of Mo target, the better the uniformity of thickness and resistance of Mo sputtered film. Moreover, when Mo target's grain size is finer and the grain boundary area per unit volume is bigger, the thickness reduction of target is more homogeneous and the sputtering film deposition is faster.
3) The microstructural differences of Mo targets have not obvious influence on grain orientation of the sputtering film.

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