Formation and investigation of thin MoS₂ films for electronics

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Abstract. The article is devoted to obtaining and studying of films of molybdenum disulfide for applications in electronics. The paper presents the results of processing images obtained using optical and atomic force microscopy of films deposited by magnetron sputtering. The structure of the substrate has shown significant influence on structure of film of MoS₂.

1. Introduction

Recently, a large number of researches have been published works about thin films molybdenum disulphide. Researchers are attracted to this material with its prospective application and useful properties for devices in the field of electronics and optoelectronics [1].

Films of molybdenum disulfide are represented a great number of monomolecular layers, which are connected in the intermolecular by Van-der-Waals forces. The sulfur and molybdenum atoms in layers are hexagonally packed crystal structure with three layers of S-Mo-S [2, 3]. Each molybdenum atom is in the center of trigonal prismatic coordination and is linked by covalent polar bonds to six sulfur atoms. Chemical formula: MoS₂. When the form of one monolayer of disulfide is meant, this formula is supplemented. Designation of the single layer of molybdenum disulfide: ML-MoS₂.

MoS₂ is a member of the transition metal dichalcogenides (TMDs) family. Atomic single-layer molybdenum disulphide (MoS₂) has a direct band gap of about 1.8 eV. MoS₂ can be served as an important addition to the place of graphene which has zero band gap, for the creation of new semiconductor elements, such as thin-film transistors [4], phototransistors [5], photodetectors [6]. The charge carrier mobility in a bulk MoS₂ is relatively high – from 200 to 500 cm²·V⁻¹·s⁻¹ [4]. Prospects for creating these devices determine interest of scientists to develop high-quality (without defect) and continuous of thin films ML-MoS₂ on a large scale and in a commercially advantageous way.

Variety of synthesis methods such as chemical vapor deposition (CVD) and physical deposition in a vacuum (PVD) are widely used for the production of TMDs, which possible to obtain grains size of ML-MoS₂ films with a diameter of 10–100 μm [7, 8]. Another method widely used for obtaining TMDs is known mechanical exfoliation. PVD methods have been produced TMD films of materials such as MoS₂ [9], WS₂ [10], WSe₂ [11]. In this paper we will examine the production of MoS₂ thin films by the method of magnetron sputtering, which could be subsequently used as a raw material for the production of ML-MoS₂ for electronics.

2. Effect of deposition factors on the structure of molybdenum disulfide

It is known, when deposited by method magnetron sputtering, the structure and properties of textured films were influenced by factors: substrate temperature [12], working argon gas pressure [13], bias voltage [14], bombarding ion energy [15], etc. Depending on these factors, different structure could be
obtained: polycrystalline, single-crystal. High crystalline quality is an important requirement to obtain the desired texture of the films for a wide range of applications. It is tested the effect of different factors on the formation and properties of films WS₂ and WSe₂ obtained by magnetron sputtering method [16]. The authors of previous work show, that the influence of temperature on the formation and structure formation of thin films molybdenum disulfide films on a silicon and graphite substrate by the CVD method [17]. In the another work [18] molybdenum disulfide film was obtained by the vacuum sputtering method, moreover, layered structures 5–20 nm thick with a texture (002) formed in the initial phase of deposition, then formed columnar structures in the later phases. Currently, it was established [19] when sputtering in a vacuum by ion-plasma methods, the influence effect of substrate temperature on the structure and its properties of obtained films MoS₂. At the different temperatures, the coating films MoS₂ formed hexagonal structures: quasi-amorphous (T < 383K), textured and polycrystalline with preferential orientation along the axes of the crystallites [100] (at T = 473–573 K and T = 773–923 K) and [110] (at T = 673–773 K) perpendicular to the surface of the substrate. Crystallites [100] (at T = 473–573 K and T = 773–923 K) and [110] (at T = 673–773 K), perpendicular to the surface of the substrate.

The authors of [20] discovered that the films began to grow with the (001) orientation, where the planes of the monolayers are preferentially parallel to the surface of the substrate. The diffraction analysis in these studies shows that the textured films (002) are obtained under conditions of high substrate temperature and a low deposition rate.

In these studies, increasing the substrate temperature can be raised mobility of the deposited atoms, that can be achieved by partial vapor ionization of the material.

In our experiment, the temperature and the type of substrate were chosen as the variable factors, as the most essential for the formation of the film.

3. Experimental
Molybdenum disulfide films were deposited by magnetron sputtering on silicon substrate (100) and glass ceramics at constant current in argon atom at two different temperatures at 200 and 300 °C. For this work for deposition we used vacuum processing unit of the Department MT-11, Bauman Moscow state technical University (BMSTU), equipped with independent ion source and balanced magnetron sputtering systems with target (MoS₂) diameter of 78 mm.

Before the deposition was performed preparation of the substrate surfaces, consisting of two stages: stage one – the substrates were cleaned in an ultrasonic bath in sequence: in alkaline solution and in ethanol (two minutes in each liquid); the stage two is the treatment of the substrate surface by the flow of argon ions from an independent ion source in a single vacuum, immediately before sputtering of the target material. The choice of these types of substrates is mainly because of they are the most commonly used types of substrates. The substrates also have a different structure: a monocristalline structure of the silicon substrate and an amorphous structure of the glass ceramics.

MoS₂ films were deposited at the same parameters on the on pre-cleaned Si substrates № 1, 2 and glass-ceramic № 3, 4. Parameters of the process of film deposition: target – MoS₂; the distance between the substrate and the target – 50 mm; working gas – argon; pressure – 9·10⁻³ mbar; substrates – Si, glass ceramic; discharge voltage – 650 V; current – 0.1 A; deposition time – 3 min. Variable parameters are shown in table 1.

| Parameter                  | № 1 Si | № 2 Si | № 3 glass ceramic | № 4 glass ceramic |
|----------------------------|--------|--------|-------------------|-------------------|
| Substrate temperature, °C  | 200    | 300    | 200               | 300               |

Table 1. Variable parameters of the process of film deposition.
4. Processing of the obtained results

The MoS₂ films were studied by using the multi-function microscope "Solver-Next" by means of atomic force microscopy, and the optical microscope from Carl Zeiss.

![Figure 1](image1.jpg)

**Figure 1.** Samples on silicon substrate: № 1: (a), (b); № 2: (c), (d). Images obtained by an optical microscope: (a), (c) low magnification; (b), (d) high magnification.

In figures 1, 2 images of coatings obtained on silicon substrate are presented for comparison itself. It is clearly that the temperature has not a significant effect on the structure and surface topography. In both cases, the surface consists of many regions of grains with a diameter of about 40 μm. Figure 2 show that the surface topography – multistage.

There are regions of grain separation on the surface of film, which show the layered structure of individual grains (figure 3). According to the results of the measurements can be predetermined that the films deposited on a silicon substrates, have the same structure: coating structures are layered on top of each other gain, each of which, also uniformly consists of individual layers. Typical grain diameter is approximately 40 μm.

Deposited coating on glass ceramic substrate also not shown dependence structure on the selected temperature range. Unlike the structure on silicon substrate, the structure of these coatings is less orderly and is not oriented preferentially parallel to the plane of the substrate. Film coated on glass ceramic shown in figure 4.

5. Results and discussion

According to the experiments and obtained analysis of microscopy images, it is possible to predetermine that the selected range of temperatures: from 200 to 300 °C, the variable factor was insignificant.

At the same time the effect of the structure of the substrate was significant. We can assume that the crystal structure of a silicon substrate to have a beneficial effect on the growth of the film and, ultimately, on its structure. Also it should be noted that the method of magnetron sputtering can be applied for coating preferably orientated MoS₂.
Figure 2. Samples on silicon substrate: № 1: (a); № 2: (b). Images obtained by atomic-force microscopy.

Figure 3. Layered of grains: three-dimensional image (a); the profile of the step (b).

Figure 4. Samples on glass ceramic: № 3: (a); № 4: (b). Images obtained by atomic-force microscopy.

6. Conclusions
MoS$_2$ coating on silicon substrate is layered structure with grain diameter about 40 μm and is oriented properly (parallel to the plane of the substrate). Because of that, we can try to attempt to get a random collection of films from the entire surface of the substrate, or in other words from many columns at a time, resulting in a nonzero percentage of the films of ML-MoS$_2$, with a size of about 40 μm. This group approach for producing films of ML-MoS$_2$ may have sufficient yield to be manufacturing applicable and commercially justified, allowing for the mass production of nanoelectronic industry.

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