AFM investigation of MoS$_2$ thin films

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Abstract. Molybdenum disulfide thin films deposited on silicon substrates by the magnetron sputtering structural features investigation by atomic force microscope results are presented.

1. Introduction

At the present time electronic properties and technologies for two-dimensional (2D) materials formation for the perspective hardware components that may be used in nanoelectronic devices, for ensuring functional and operational characteristics of electronic systems further increasing are widely and actively studied. Molybdenum disulfide (MoS$_2$) with one or more molecular layers thickness is most widely studied. MoS$_2$ monomolecular layers is estimated 100–480 cm$^2$ (V s)$^{-1}$ charges mobility. As distinct from graphene, the monomolecular layer of molybdenum disulfide possess 1.8 eV direct bandgap and it is the direct-band-gap semiconductor, more thicker MoS$_2$ films demonstrate the indirect semiconductor properties with a bandgap from 1.3 to 1.8 eV, depending on a thickness [1]. A much more number of the modern works performed by different authors, where engineering development samples of electronic devices based on MoS$_2$ films were prepared, demonstrate the prospects of its application in field effect transistors (FET), light-activated transistors, organic electronics and other devices. Engineering development devices are created in a laboratory environment, generally using molecular layers mechanical splitting and separation from bulk crystal methods.

The method of mechanical splitting MoS$_2$ layers is suitable for monolayer 2D materials laboratory samples creation, which is widely known as «Scotch method», is inapplicable in industrial production. Therefore, today a lot of researchers solve the concerning technology problem of MoS$_2$ films' formation on large area substrates renewable at industrial scale. For these purposes the following possible methods are investigated: chemical vapor deposition (CVD), atomic layer deposition (ALD), the technology based on the mechanical printability by transfer elements from a stamp on a substrate surface [2], ion sputtering target methods in vacuum and others. In this work the research results of MoS$_2$ thin films forming by magnetron sputtering cathode target in the argon environment on a silicon substrates are presented.

MoS$_2$ films surface researches were conducted by atomic force microscopy (AFM) methodic. Films' structural features analysis consisted in a surface texture scanning by the AFM method and processing received images by IA-P9 program reference techniques for the identification films formation technological modes influence on a films surface features.
2. Experimental

MoS$_2$ thin films were deposited at vacuum equipment fitted with a balanced magnetron sputtered systems. Before films deposition liquids and ions cleaning substrates surface treatment were produced.

For the magnetron sputtering parameters influence on a films structural features researching, ten samples with a MoS$_2$ thin films were deposited on silicon substrates at the different conditions. Table 1 shows the main technological parameters.

| Sample number | Argon pressure, Pa | Discharge power, W | Substrate bias, Volt | Substrate heating temperature, °C | Deposition time, min |
|---------------|-------------------|--------------------|---------------------|----------------------------------|---------------------|
| 1             | 7.50x10$^{-1}$    | 52                 | 20                  | 200                              | 65                  |
| 2             | 7.30x10$^{-1}$    | 45                 | 30                  | 200                              | 60                  |
| 3             | 4.50x10$^{-1}$    | 30                 | 20                  | 200                              | 30                  |
| 4             | 7.50x10$^{-1}$    | 45                 | 27                  | 200                              | 75                  |
| 5             | 4.40x10$^{-1}$    | 45                 | 22                  | 250                              | 32                  |
| 6             | 1.00              | 30                 | –                   | 250                              | 30                  |
| 7             | 1.00              | 30                 | –                   | 250                              | 45                  |
| 8             | 6.00x10$^{-1}$    | 30                 | –                   | 250                              | 110                 |
| 9             | 1.00x10$^{-2}$    | 10                 | –                   | 250                              | 30                  |
| 10            | 6.10x10$^{-1}$    | 30                 | –                   | 300                              | 60                  |

Three-dimensional surface topography AFM-images gives nano-dimensional data about films structure, including a roughness, imperfections, amorphous and crystalline phases, nucleation specifics and a mode of growth [3, 4].

Films surfaces AFM-scanning at contact mode was carried out at atomic-force microscope "Solver-NEXT" designed by "NT-MDT" company. Samples have been attached to a metal holder, and then on a surface of each sample the area in the size 30×30 μm has been in a random way selected and scanned. The real thickness of the received films was defined throw the step prepared by masking part of a surface during deposition, and for samples with the maximum deposition time measured up to 2 microns.

Surface texture scanning results visualization was received as topographic maps and 3D images. On topographic maps sections along which surface profiles were under construction were selected. With IAP9 software using the roughness statistical parameters of the chosen profiles was received.

| Sample number | № 6 | № 7 | № 8 |
|---------------|-----|-----|-----|
| Height of the surface lowermost points $h_{min}$, nm | -2,444 | -8,237 | -16,326 |
| Height of the surface uppermost points $h_{max}$, nm | 2,453 | 6,214 | 21,552 |
| The maximum overall of heights $R_y$, nm | 4,897 | 14,451 | 37,878 |
| Average square roughness $R_s$, nm | 0,868 | 2,365 | 7,822 |
| Average arithmetical roughness $R_a$, nm | 0,691 | 2,092 | 6,417 |
| Quantity of peaks | 19 | 24 | 28 |
| Distance between peaks, μm | 1.58 | 1.25 | 1.07 |
For the samples number 6, 7 and 8, prepared at the identical target sputtering conditions (power discharge 30 W, substrate temperature 250 °C) but various deposition times (30, 45, 110 minutes), has been realized surface profiles additional handling at the most typical for all scans regions. The prepared profiles were used for average distance between peaks on a surface calculation for the purpose of its planarity estimation. Roughness parameters and average distances between profile peaks calculated values are represented in table 2.

3. Results and discussion

As the received results displayed, with the deposition time increasing and the MoS$_2$ films thickness growth the surface roughness rise appreciably (essentially several times) that can testify about three-dimensional structure growth character tendencies reinforcement. For small deposition time, at small film thickness higher surface planarity is observed for the sample number 6, and the average distance between peaks of its profile essentially higher, than at samples numbers 7, 8.

The space of roughness $R_a$ values for various technological conditions depending on MoS$_2$ films deposition time is presented at Figure 1. It is possible to mark the most essential influence of a preheating substrate temperature on a deposited films roughness magnitude, and with a temperature reducing roughness essential growth is observed. With the increasing deposition time, the steady tendency to roughness growth, for all another technological conditions, is observed too.

For the samples received at temperature 250 °C and deposition time 30 min, changing argon pressure (from 0.01 to 1 Pa) and discharge power (10 W, and 30 W) had no such significant effect on a roughness, as in a case with such influencing parameters, as temperature and deposition time.

![Figure 1. Deposition time influence on a roughness at a different technological condition.](image)

Figure 2 shows films numbers 5 and 6 AFM-images and also surface heights distribution histograms respectively for these films, calculated for all scanned area 30 μm × 30 μm. It is revealed that surface heights distribution for these samples differ substantively.
So, for a sample number 5 that was formed at higher discharge power and smaller pressure in comparison with a sample number 6, the considerable positive asymmetry of distribution is observed (figure 2(a)). At the same time the sample number 6 has smoother surface (figure 2(b)) and there is a slight negative asymmetry at almost symmetrical distribution. It allows making a conclusion that discharge power takes effect on a roughness and structural features of the formed MoS$_2$ films.

**Figure 2.** AFM-images and distribution histograms of a films surface heights: (a) – sample 5, (b) – sample 6.

**Conclusions**

This work received results allows making some outputs:

1. Substrate temperature, and also discharge power and argon pressure has significant effect on a structural features MoS$_2$ thin films.

2. With increase deposition time the increase films surface roughness that can be linked with a growth features changes at increase film thickness and surface influence at substructure lowering, characteristic growth for an initial stage is observed.

3. AFM investigation method, taking into account with the known features of MoS$_2$ anisotropic structure can be used for the analysis of molybdenum disulfide thin films.

**References**

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