CVD growth of two-dimensional MoS_2 depending on the location of the SiO_2 substrates

E. I. Zakharkina, A. A. Semenova, E. F. Boiakinov, P. V. Vinokurov, and S. A. Smagulova

Citation: AIP Conference Proceedings **2041**, 040001 (2018); doi: 10.1063/1.5079366
View online: https://doi.org/10.1063/1.5079366
View Table of Contents: http://aip.scitation.org/toc/apc/2041/1
Published by the American Institute of Physics

CVD Growth of Two-Dimensional MoS_2 Depending on the Location of the SiO_2 Substrates

E.I. Zakharkina[a)], A.A. Semenova, E.F. Boiakinov, P.V. Vinokurov and S.A. Smagulova

North-Eastern Federal University, 58 Belinsky str, Yakutsk, 677000, Russian Federation

[a)]Corresponding author: evdokiya_21@mail.ru

Abstract. Transition metal dichalcogenides, like MoS_2, were attracted attention due to their optoelectronic properties. But fabrications of such materials still a challenge task. In this work, chemical vapor deposition (CVD) was used to synthesize two-dimensional MoS_2 from S and MoO_3 powders on SiO_2. Lateral size of domains were up to 80 mkm and thickness of single-layered MoS_2 was 0.9 nm. The structural and optical properties of the obtained two-dimensional MoS_2 are studied. We find that the formation of MoS_2 domains with different size is dependent on location of the SiO_2 substrates.

INTRODUCTION

After the discovery of graphene, great progress has been made in obtaining other two-dimensional materials. Today, such materials, like the class of transition metal dichalcogenides, in particular molybdenum disulphide (MoS_2) shows great interest [1,2]. Molybdenum disulphide is one of the most promising semiconductor materials for nanoelectronics, optics and spintronics due to its unusual electronic and optical properties [3]. Multilayer MoS_2 is a semiconductor with an indirect 1.2 eV band gap. However, the most interesting properties of MoS_22 appear in the study of single-layer MoS_2, which is a 1.8 eV direct band gap semiconductor [4].

At present two-dimentional MoS_2 is obtained by mechanical exfoliation, electrochemical intercalation of lithium ions, molecular beam epitaxy, atomic-layer deposition and other methods [5]. But the most promising method is chemical vapor deposition (CVD), which makes it possible to obtain uniform two-dimensional MoS_2 films of large area, which is an urgent task for industrial production [6].

METHODS

For the growth of MoS_2, silicon with an oxide film of SiO_2 300 nm in thickness was used as substrates. Before the growth process, the surfaces of the substrates were pretreated in an ultrasonic bath with acetone and isopropyl alcohol for 5 minutes to remove contaminants. To remove organic bonds from the surface, the substrates were treated in a sulfuric peroxide solution in a ratio of 1:3, followed by washing in deionized water. The substrates were then processed in an oxygen plasma for 5 minutes at a power of 150 W.

The synthesis of molybdenum disulphide was carried out by the CVD method in the three-zone ceramic furnace Nabertherm 80/750/RS, schematically depicted in Fig. 1. In a quartz tube 5 cm in diameter, crucibles with precursors and samples were placed at a certain distance from each other as shown in Fig.1 This tube was subsequently introduced into a large ceramic tube of the furnace. The starting precursors for the synthesis were MoO_3 and sulfur powders of Sigma Aldrich in a weight ratio of 1:13. The substrates on which MoS_2 was grown were located at different distances from the precursor MoO_3. To reduce the amount of evaporated MoO_3, the crucible was covered with a silicon substrate. For the controlled synthesis, parameters were chosen: temperature, time, pressure, argon supply rate, initial precursor concentrations and location of substrates relative to precursors. Synthesis process includes:

1. The pre-vacuum pump is evacuated to 10-5 bar for 1 hour.

2D Systems of the Strong Correlated Electrons: From Fundamental Research to Practical Applications
AIP Conf. Proc. 2041, 040001-1–040001-4; https://doi.org/10.1063/1.5079366
Published by AIP Publishing. 978-0-7354-1767-0/$30.00

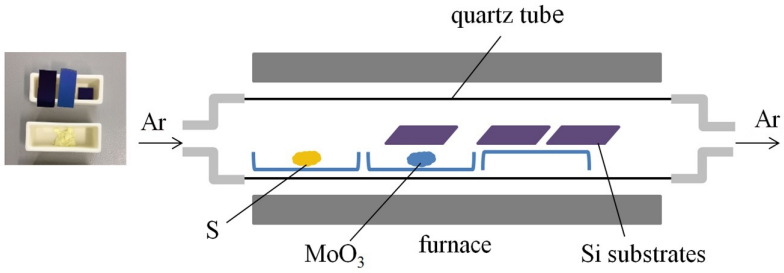


FIGURE 1. Schematic illustration of the CVD growth setup of SiO_2

2. Inert gas (Ar) is fed into the chamber at a flow rate of 100 cm^3/min for 1 hour to a pressure of 1 atm in the chamber when the temperature reaches 300°C.

3. The chamber is heated to a synthesis temperature of 700°C. 4. The growth of MoS_2 on SiO_2 occurs in the Ar flow at a rate of 10 cm^3/min for 30 minutes at 700°C.

5. After synthesis, the chamber was naturally cooled to room temperature in the Ar stream to 300 cm^3/min.

Optical images were taken with an optical microscope Nikon eclipse LV100. The morphology of the surface of MoS_2 films grown by CVD on the SiO_2 substrate was studied by scanning probe microscope AFM / STM "SolverNext" and scanning electron microscope JEOL JSM-7800F. Measurements of the Raman spectra of MoS_2 were carried out at room temperature using NT-NTRA SPECTRA from NT-MDT. The spectra were excited by a semiconductor laser with an excitation wavelength of 532 nm. Elemental analysis of the composition of grown MoS_2 films was carried out by Oxford Instruments Energy-Dispersive Spectroscopy (EDS).

RESULT AND DISCUSSION

Optical measurements showed the growth of domains of MoS_2 on the SiO_2 surface in the form of triangles (Fig. 2). Depending on the location of the substrates relative to the MoO_3 precursor, domains with different lateral sizes were obtained. Samples were located 5-7 cm from the precursor MoO_3, domains were 10-20 mkm. With decreasing the distance between samples and precursor domain size was increased up to 40 mkm. The largest MoS_2 triangles were obtained, when samples located directly above the precursor. According to our results, larger flakes were grown near Mo precursor because of higher concentration MoO_3 and different gas flow through the surfaces of substrates.

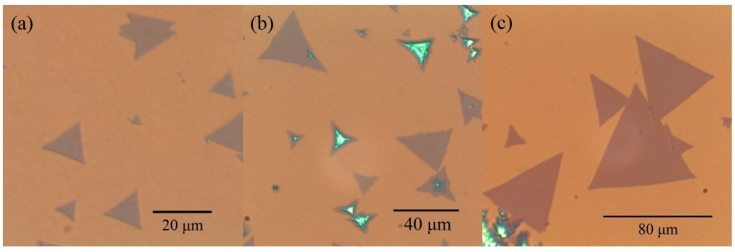


FIGURE 2. Optical images of domains grown at different distances from the precursor MoO_3: (a) more than 5 cm, (b) more than 2 cm, (c) directly above the precursor

AFM measurement showed 0.9 nm thickness of one layer of molybdenum disulphide (Fig. 3). Also, the surface of the obtained MoS_2 was studied, where the presence of defects in the center of some domains was shown, and overgrown fragments of the same material on the edges of the domains were found.

Figure 4 shows the Raman spectra of MoS_2 with two most intense Raman peaks in the 380 and 400 $cm^{-}1$ region were observed. The frequency difference between these peaks allowed to determinate the number of layers of grown

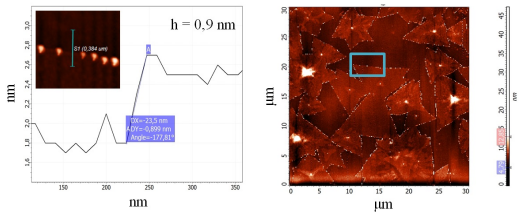


FIGURE 3. AFM image of a single-layer MoS_2 domains on the SiO_2 surface and the height profile along the line S1

MoS_2. For single-layer MoS_2, the frequency difference between the peaks was $\Delta\nu = 19\ cm^{-1}$, for the bi-layer $\Delta\nu = 21\ cm^{-1}$ and for the multi-layer $\Delta\nu = 25\ cm^{-1}$.

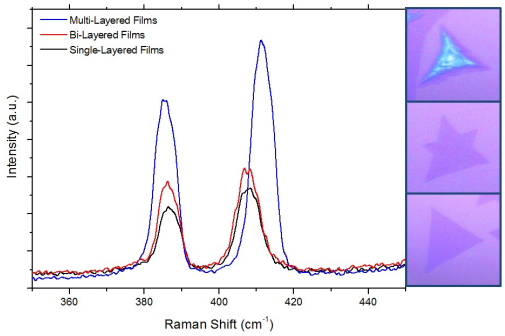
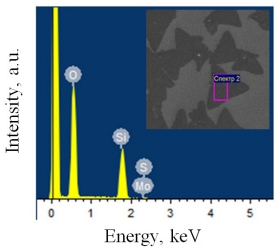


FIGURE 4. Raman spectra of synthesized single-layer, two-layer and multilayer MoS_2

The data of the energy dispersive X-ray spectral microanalysis, which is shown in Fig. 6, made it possible to determine the elemental composition of the samples-molybdenum (0.19 at.%), sulfur (0.48 at.%), silicon (28.41 at.%), oxygen (70, 91 at.%). It should be noted that the spectral peaks of molybdenum and sulfur are located at values of about 2.3 keV and have only minor deviations with respect to each other, which made it difficult to identify this elements. Nevertheless, it was found that the atomic sulfur content is twice the atomic molybdenum content.



Element	Atomic %
O	70,9
Si	28,4
S	0,5
Mo	0,2

FIGURE 5. The line-scan EDX spectrum of a selected region from the STEM image (marked by a pink color rectangle)

CONCLUSIONS

Single-layer MoS_2 with a thickness of up to 0.9 nm and lateral domain sizes up to 80 mkm was obtained on a SiO_2 substrate by proposed CVD method. The domains with the largest lateral dimensions were obtained for samples were located less than 5 cm from the MoO_3 precursor. It was found that with increasing distance the size of MoS_2 domains decreases. According to our results, larger flakes were grown near Mo precursor because of higher concentration MoO_3 and different gas flow through the surfaces of substrates.

ACKNOWLEDGMENTS

The reported study was funded by RFBR according to the research project N 18-32-00730.

The reported research was funded by Russian Foundation for Basic Research and the government of the region of the Russian Federation, grant N 18-42-140005.

REFERENCES

[1] K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, Y. Zhang,S. V. Dubonos, I. V. Grigorieva, A. A. Firsov *Science*, **306(5696)**, 666–669 (2004).
[2] B. Radisavljevic, J. Brivio,V. Giacometti, A. Kis *Nature Nanotechnology* **6**, 147–150 (2011).
[3] X. Duan et al. *Chem. Soc. Rev.* **44(24)**, 8859–8876 (2015).
[4] A. Splendiani, L. Sun, Y. Zhang, T. Li, J. Kim, C.-Y. Chim, et al. *Nano Lett.* **10**, 1271–1275 (2010).
[5] X. Li, H. Zhu *Chem. Soc. Rev.* **1(1)**, 33–44 (2015).
[6] Q. Ji, Y. Zhang, Y. Zhang, Z. Liu *Chem. Soc. Rev.* **44(9)**, 2587–2602 (2015).