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NANOSIZED VERTICAL NANOSHEETS MADE OF MOLYBDENUM DISULPHIDE: ELECTRICAL AND OPTOELECTRONIC PROPERTIES

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Abstract. In the paper, the results of the studies in the electrical and optoelectronic properties of vertical sheets made of MoS_2 have been presented for the first time. These objects are characterized by a high surface area, exposed edges, reasonable carrier mobility values and high light absorptance. Samples with an average size of about 150 nm were grown by the one-stage metal-organic chemical vapor deposition (MOCVD) technique. Vertically oriented MoS_2 sheets were investigated using the scanning electron and X-ray photoelectron microscopy, the X-ray diffraction and Raman spectroscopy. VI characteristics of the samples were obtained as well. Optoelectronic properties of the samples were studied using an argon laser (operates at a wavelength of 513 nm) with a mechanical light modulator. An analysis of the obtained results allows us to state that the studied V-MoS_2 sheets should be considered as a very promising material for optoelectronics needs.

Keywords: chemical vapor deposition, molybdenum disulphide, vertical nanosheet, optoelectronic and electrical properties

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ЭЛЕКТРИЧЕСКИЕ И ОПТОЭЛЕКТРОННЫЕ СВОЙСТВА НАНОРАЗМЕРНЫХ ВЕРТИКАЛЬНЫХ НАНОЛИСТОВ, ИЗГОТОВЛЕННЫХ ИЗ ДИСУЛЬФИДА МОЛИБДЕНА

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Аннотация. В статье впервые представлены результаты исследования электрических и оптоэлектронных свойств вертикальных листов, изготовленных из дисульфида молибдена MoS_2 (V-MoS_2). Этим объектам свойственны большая удельная поверхность, открытые края, разумные значения подвижности носителей и высокий уровень поглощения света. Образцы со средним размером около 150 нм были выращены методом одностадийного ХОГФ при использовании металлорганических исходных соединений (MOCVD). Листы V-MoS_2 изучены методами сканирующей электронной и рентгеновской фотоэлектронной микроскопии, рентгеновской дифракции и спектроскопии комбинационного светорассеяния. Получены вольтамперные характеристики образцов.

Оптоэлектронные свойства V-MoS_2 исследованы с помощью аргонового лазера (длина волны – 513 нм) с механическим модулятором света. Анализ полученных результатов позволяет утверждать, что изученные листы V-MoS_2 следует рассматривать как весьма перспективный материал для нужд оптоэлектроники.

Ключевые слова: ХОГФ, дисульфид молибдена, вертикальный нанолист, оптические и электрооптические свойства

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Introduction

Among the family of transition metal dichalcogenides (TMDs), molybdenum disulphide MoS_2 is one of the most extensively studied materials due to attractive properties of its thin films, such as possibility to transform band structure from an indirect bandgap to a direct one by decreasing their thickness from bulk to a single layer [1, 2]; high room-temperature carrier mobility in MoS_2 (was measured to be about 200 $cm^2 \cdot V^{-1} \cdot s^{-1}$) with a large switching on/off ratio exceeding 10^8 value, strong interaction with light and low energy consumption [1 – 5]. Consequently, MoS_2 has attracted considerable interest as a promising candidate for manufacturing enhanced transistors, sensors, photodetection and electronic displays [5 – 8]. In addition, MoS_2 has a promising outlook in the fields of solar cells, energy storage, energy conversions and catalytic applications. For example, MoS_2 can be used as a highly efficient electrocatalyst for hydrogen evolution reaction [9 – 14].

Most of the papers published during the last decade have been devoted to formation of mono- or few- layers planar MoS_2 structures deposited on the surface of the substrate (mainly sapphire or silicon oxide). However, recently the deposition of vertically aligned sheets of MoS_2 has been achieved and remarkable interest appeared in them due to their specific features including maximum surface area and extensively exposed edges [15 – 17].

The vertical MoS_2 sheets have a complicated structure that has many dongle bonds, in comparison to the layers grown horizontally on the substrate surface. Although many research groups have reported an formation of the MoS_2 vertical nanosheets, their electrical and optoelectronic properties have not been fully studied yet.

The purpose of this work was to make nanosized vertical sheets of MoS_2 (V-MoS_2) and to study their electrical and optoelectronic properties.

Materials and methods

The deposition process was carried out at a low pressure in a hot-wall horizontal tube reactor with a diameter of 56 mm and a length of about 300 mm made of quartz. $Mo(CO)_6$ powder and H_2S gas were used as precursors for metal-organic chemical vapor deposition (MOCVD) to grow MoS_2 films. The molybdenum containing the precursor $Mo(CO)_6$ was introduced into the deposition chamber from the evaporator maintained at the temperature of 30 °C by using argon as a carrier gas. To ensure the complete transfer of the precursor into the reactor, the vapor transport lines were maintained at about 120 °C. The total pressure in the reaction chamber was set to approximately 70 Pa, the substrate temperature was approximately 550 °C and the deposition time was 30 min. The substrates (silicon wafer, silicon wafer with deposited 100 nm SiO_2 film, fused quartz) were cleaned in acetone, alcohol and deionised water for 10 min.

The morphology and composition of the deposited films were studied with the use of scanning electron microscopy (Supra 55 VP with WDX and EDX spectrometers). X-ray photoelectron spectroscopy (SPECS HAS 3500) was used for chemical analysis. The presence of crystalline phases was investigated using X-ray diffraction (Super Nova Dual Wavelength

(Agilent Technology), Cu K_α (λ = 1.5405 Å)) and Raman spectrum was measured by Raman spectrometer (Horiba 800).

Silver electrodes were deposited on the samples by electron beam evaporation using high vacuum system (10^{-6} Torr) and electrical properties were measured by Keithley 237 under pressure of about 10^{-4} Torr in Janis cryostat. Voltage-current (VI) measurements were carried out on three samples (repeated 5 times for each sample), and then the measurement results were averaged. Optoelectronic properties were measured using an argon laser (green light) with mechanical chopper.

Results and discussion

Structure and composition. The scanning electron microscopy (SEM) images of the deposited film made on the surface of SiO_2/Si substrate are presented in Fig. 1. As can be seen, the film consists of sheets grown perpendicular to the surface of the substrate. The sheet sizes vary and their average value is about 150 nm. The cross-section of the layer is shown in the inset of the same figure and it clearly shows the vertical growth of the sheets with a height of about 250 nm. Films deposited on the silicon and quartz substrates were characterized by similar morphology without any noticeable differences.

Crystal structure of the films was studied using powder X-ray diffraction (PXRD). As Fig. 2 suggests, all reflections can be attributed to the pure hexagonal phase of MoS_2 with the following lattice parameters:

$$a = 3.161 \text{ Å and } c = 12.299 \text{ Å}$$

(standard file JCPDS No. 37-1492) and diffraction peaks from crystalline impurities were not observed. Since the X-ray spectrometer had “powder geometry”, it was possible to observe reflections only from planes parallel to the substrate. In this connection, it seems highly probable that the strong reflection (00L) comes from the horizontal layer, while reflections (100) and (101) can refer to vertical sheets. Using the Scherer formula, the thickness of the horizontal layer was calculated from the FWHM value of the diffraction line (002). The minimum thickness of the horizontal layer was found to be about 37 nm.

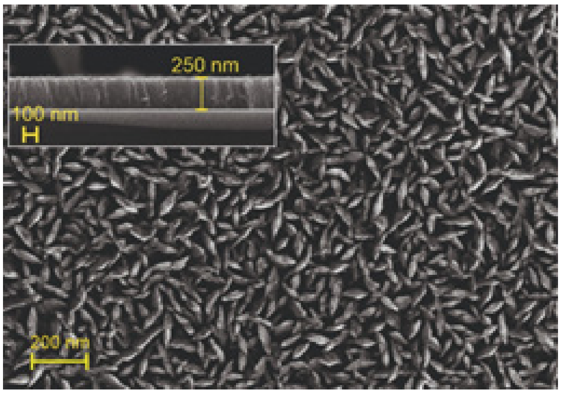

Fig. 1. SEM image (top-view) of vertical MoS_2 nanosheets: their sizes vary and the most are around 150 nm. Inset: the cross section of the objects

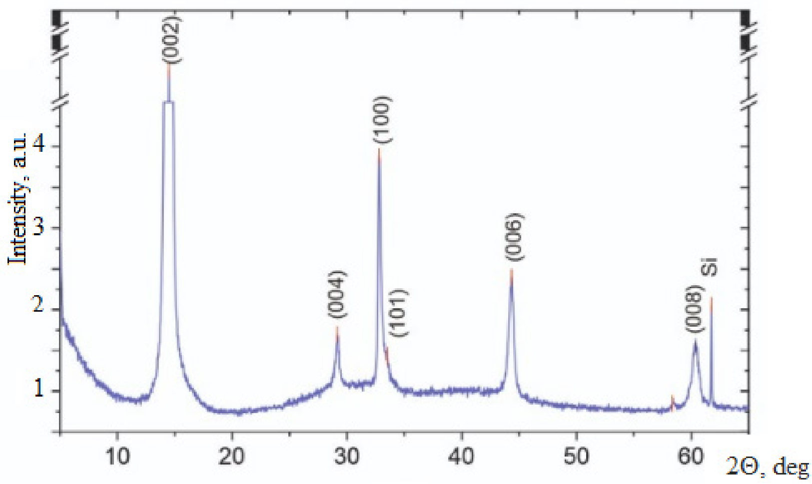

Fig. 2. PXRD pattern of the MoS_2 film with vertical nanosheets on the silicon oxide substrate with peaks indexed

The Raman spectroscopy is widely used to study crystal structure, the quality of MoS_2 substance, the number of monolayers and texture of MoS_2 films. Two strong characteristic Raman modes E_{12g} and A_{1g} of MoS_2 were observed in the Raman spectra of the deposited films with vertical nanosheets at 381 cm^{-1} and 407 cm^{-1}, corresponding to in-plane vibration of molybdenum and sulfur atoms, and out-of-plane vibration of sulfur atoms, respectively (Fig. 3). Both modes show a red shift of about 1 cm^{-1} comparing to the values typical for the bulk MoS_2 probably due to strain in the films. The frequency difference between the E_{12g} and A_{1g} Raman modes is about 26 cm^{-1}, which indicates the presence of around seven or more layers in the MoS_2 nanosheets.

The energy dispersive X-ray (EDX) and X-ray photoemission (XP) spectra of the film consisting of vertical sheets have signals from Mo, S, C and O atoms only. It seems very likely that the presence of oxygen and carbon is caused by their adsorption from the surrounding atmosphere. The composition of the MoS_2 film is close to stoichiometric and it has been separately confirmed by the results of XP and EDX spectroscopies (the S/Mo ratio is about 2.01). High resolution XP spectroscopy analysis was carried out to investigate the chemical states of Mo atoms. The deconvolution of the Mo 3*d* core spectrum by peak fitting reveals two Mo 3*d* doublets (see Fig. 4). The Mo-atom signal mainly arises from Mo $3d_{5/2}$ (228.9 eV) and Mo $3d_{3/2}$ (232.1 eV) characterized the molybdenum Mo^{4+} sulphide components. The small doublet at lower binding energy (233.0 and 230.2 eV) can be related to defects or 1T phase of MoS_2, while the doublet at higher energy (233.4 and 235.7 eV) corresponds to higher oxidation states (Mo^{5+}) and (Mo^{6+}) due to the presence of molybdenum oxide and defects.

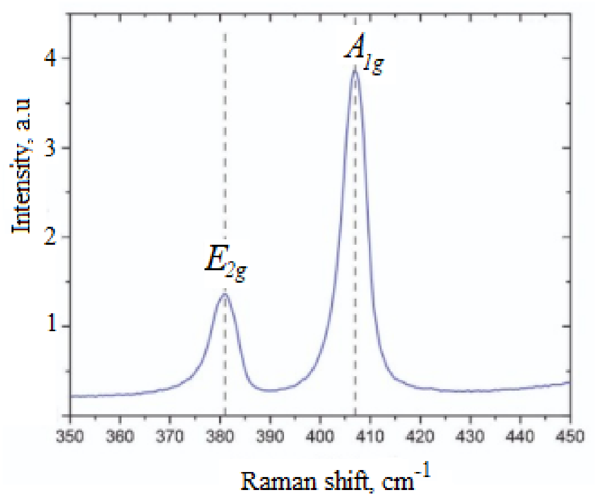

Fig. 3. Raman spectrum pattern of MoS_2 film with vertical nanosheets on silicon oxide substrate

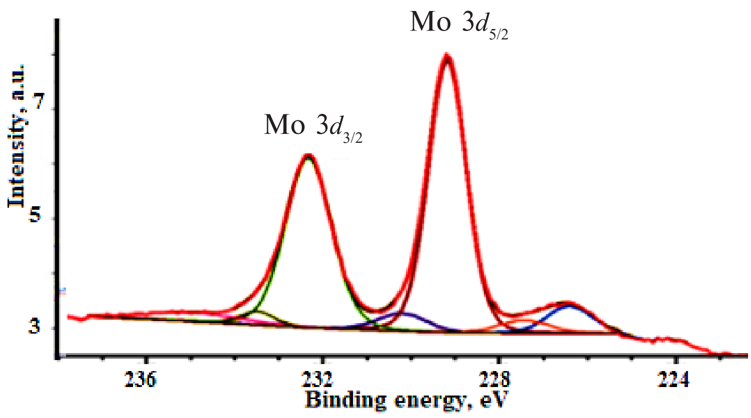

Fig. 4. XP spectrum of MoS_2 layer, high resolution spectrum of Mo 3*d* components with fitting (the red line is an experimental datum and the black one is a fitting result)

Thus, the results of the XRD analysis suggest that the vertical nanosheets are formed not directly on the substrate surface, but on the surface of the horizontally grown MoS_2 layer with a certain thickness. That issue has already been raised in our Ref. [18].

Electrical and optoelectronic properties. Silver films were used as the contact layer to the MoS_2 ones (the films were deposited on the SiO_2/Si substrates) since the ohmic nature of the contact can theoretically be expected considering the fact that Ag and MoS_2 have work function values of 4.5 eV and 5.2 eV, respectively. Voltage-current (VI) relationships measured for vertical sheets under vacuum are shown in Fig. 5. From this figure we notice that they are not linear.

Several models including the Schottky emission, direct tunneling, the Poole – Frenkel emission, and space-charge-limited-current (SCLC) state [19] were used for fitting them with experimental curves, and it was found that the Lampert theory of SCLC could be successfully used to explain the current behavior. As can be seen from Fig. 5, the VI curve plotted in the ($\lg V - \lg I$) coordinates has four distinct regions: ohmic where $I \sim V$ (region I), the Mott – Gurney's law $I \sim V^{3/2}$ (IV), and the trap-filled limit voltage (VTFL) (II). The III region corresponds to the transition from the trap-limited conduction to the trap-free one. V_{ON} and V_{TFL} are the voltage values for the transition from ohmic conducting to the Mott – Gurney's law and from trap-filled conducting to the Mott – Gurney's law, respectively.

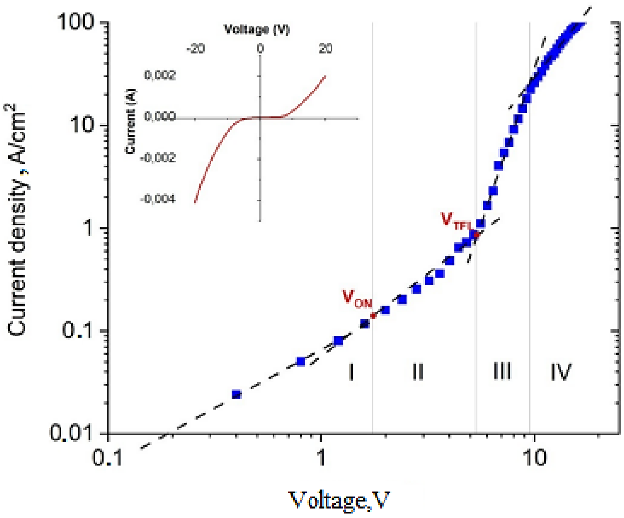

Fig. 5. Current density plotted as a function of the applied voltage on the logarithmic scale, insert: the IV region for vertical sheets on linear scale. All measurements were made under vacuum

Using the SCLC method made it possible to calculate a lot of important transport parameters including the carrier mobility, the concentration of free charge carrier and the trap density. Based on the SCLC model for thin films and using the procedure described in Ref. [19], the electrical properties of vertical sheets of MoS_2 were calculated and tabulated.

Molybdenum disulphide MoS_2 is a material with moderated carrier mobility and the highest values usually correspond to the monolayers (the maximum value reported is about 200 cm^2/(V·s) [1]). It should be noted that the mobility value of about 45 cm^2/(V·s) measured in this work for vertical sheets is within the typical range. However, after filling the traps, a noticeable increase in the mobility is detected by more than an order of magnitude (region IV), demonstrating a strong effect of defects on mobility in MoS_2. Such value has to be considered as rather high taking into account the low deposition temperature (550 °C) of MoS_2 nanosheets and the complicated structure of the sheets. To confirm this high value of the carrier mobility time-dependent photoresponses were measured by exposing vertical sheets to the argon laser light (513 nm) with 10 V bias. Time responses were measured by real-time CW laser on/off using a variable speed-controlled mechanical chopper. The fulling time t was estimated by fitting with exponential function

$$y - y_0 = A \cdot \exp\left[(x_0 - x)/t\right], \quad (1)$$

where x_0 and y_0 are the initial values, i. e. the time and current values for the moment when the light was turned off.

As can be seen from Fig. 6, the measured values of the rise time (0.65 ms) and the fall time (0.69 ms) are quite small and agree well with the relaxation time calculated using the SCLC method (see the Table).

Table

Electrical properties of MoS_2 vertical sheets calculated from SCLC curves (see Fig. 5)

Parameter	Unit	Curve region used	Parameter value
Carrier mobility	$cm^2/(V{\cdot}s)$	II	31.4
		IV	475.6
Carrier concentration (in thermal equilibrium)	cm^{-3}	Border I – II	1.0759e+16
Density of traps		Border II – III	6.673e+16
Dielectric relaxation time	s	II	1.41e–2
		IV	7.30e–4

Footnotes. 1. The calculations were performed using the procedure given in Ref. [19]. 2. The dielectric relaxation time was obtained using the calculated carrier mobility as the base.

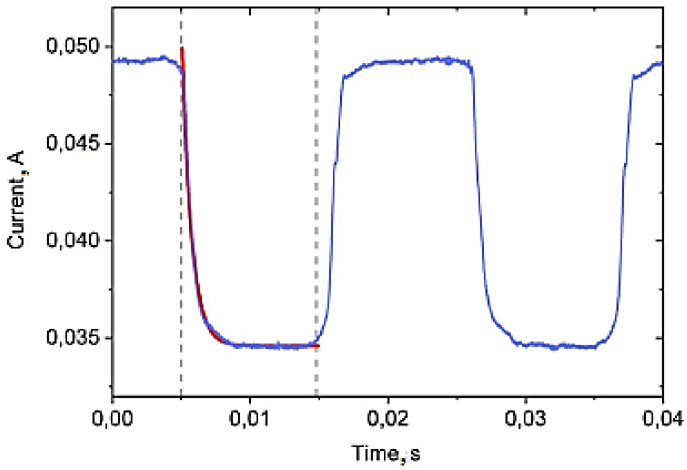

Fig. 6. Photoresponse curve of vertical nanosheets measured using a CW laser with mechanical chopper. The fulling time was estimated by fitting with exponential function (1)

Summary

The results obtained allow us to conclude that V-MoS_2 nanoscale vertical sheets should be considered as a promising material for the needs of optoelectronics. The study carried out has been demonstrated that MoS_2 nanosheets are able to provide high mobility and fast optoelectronic response.

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