Crossref
DOI: 10.31643/2026/6445.38
Engineering and technology

Morphological and Crystallographic Investigation of CVD-Grown MoS_2

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Received: *February 14, 2025*
Peer-reviewed: *April 8, 2025*
Accepted: *June 9, 2025*

ABSTRACT

This paper presents a study of the structural characteristics of a promising MoS_2-based material obtained by chemical vapor deposition (CVD). Optimization of the synthesis process to obtain the desired structure is also presented. The optimal parameter for the synthesis of CVD MoS_2 crystals was found to be the maximum sulfurization temperature of 780 °C with an exposure time of about 15 minutes, the heating temperature of the sulfur source zone of 250 °C, the distance between the sulfur and molybdenum sources of 25 cm, and the distance between the molybdenum source and the substrate was 1.5 cm. The morphology and elemental composition of the obtained samples were studied using scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS). Using SEM, it was revealed that MoS_2 crystals are formed in a triangular shape and are evenly distributed over the surface of the substrate. The maximum sizes of crystallites reach 6 microns. EMF mapping of crystallites confirmed the homogeneous distribution of molybdenum and sulfur in the structure, revealing only minor variations in composition at the grain boundaries. The quality and quantity of the sample layer were studied using Raman spectroscopy. The results showed two characteristic peaks (vibrational modes E_{2g}^1 and A_{1g}) of nanoscale MoS_2. The peaks have a sharp shape and are located at a distance of ≈20.9 cm^{-1}, which may indicate the high quality of the crystal structure of the obtained crystallites. The results obtained emphasize the effectiveness of the chosen approach and the importance of the work for the development of 2D materials technologies.

Keywords: Molybdenum disulfide, CVD synthesis, 2D materials, Raman spectroscopy, morphology.

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Introduction

Molybdenum disulfide (MoS_2) is a layered material possessing a set of unique properties—including semiconducting, optical, mechanical, and catalytic characteristics—that make it a subject of intensive research. Its electronic properties are closely related to its structural phase. In its monolayer form, the semiconducting 2H-MoS_2 phase exhibits a direct bandgap of approximately 1.8–1.9 eV, making it a promising candidate for applications in field-effect transistors and photodetectors [1]. In contrast, the metallic 1T-MoS_2 phase broadens its functionality in catalytic and energy systems [2].

MoS_2 is noted for its high mechanical strength, low friction coefficient, and pronounced catalytic activity, particularly in the hydrogen evolution reaction (HER) [[3], [4]]. These attributes have stimulated their application in tribology and renewable energy technologies [[5], [6]]. In the field of nanoelectronics and optoelectronics, MoS_2 has been integrated into field-effect transistors, light-emitting diodes, photodetectors, and solar cells [7].

Its high absorption and emission efficiency facilitate the development of fast and sensitive photonic devices [[8], [9]]. Furthermore, its catalytic properties can be tailored through surface modification to enhance device performance [3]. MoS_2's tribological advantages—such as high wear resistance—make it an effective solid lubricant or protective coating [5], while in sensing applications, MoS_2 demonstrates selective adsorption of target molecules, which is advantageous for gas and biosensors [[10], [11]].

In recent years, efforts have been made to expand MoS_2's application potential via functionalization. Various surface modification techniques have been shown to tune MoS_2's physicochemical properties, improving its environmental stability and performance in devices [[12], [13], [14]]. The creation of hybrid structures by combining MoS_2 with other two-dimensional (2D) materials, such as graphene or metal oxide nanostructures, has been shown to enhance their electrical conductivity and catalytic activity [[15], [16], [17], [18]]. In addition, chemical treatments that increase the density of catalytically active sites are essential for optimizing electrochemical performance [[19], [20]].

Different morphologies and structural qualities of MoS_2 have been obtained using different synthesis routes. Among these, chemical vapor deposition (CVD) is frequently selected because this method is scalable as well as offers layer thickness and uniform control [21]. Mechanical exfoliation [22], hydrothermal synthesis [14], laser ablation, and other methods, such as ultrasound-assisted or biological synthesis [[23], [24], [25]], still have a role in meeting other needs. However, some issues prevent practical deployment. Phase instability is one of the bigger ones. However, the 1T- MoS_2 phase is more active but undergoes irreversible conversion to the stable 2H- MoS_2 phase under normal and ambient conditions [21]. The Stabilising it chemically or structurally is an ongoing challenge. One issue is reproducibility, as growth through CVD is highly dependent on the experimental parameters such as temperature, position of precursor and gas flow dynamics [[26], [27]].

Devices suffer performance limitations as well. In general most basal planes of MoS_2 are less catalytically active and its conductivity is not always high enough for demanding electronic applications. But that progress has been made by forming nanostructures or integration of MoS_2 with conductive frameworks such as carbon nanotubes or graphene [[28], [29], [30]]. CVD has recently been achieved in large areas on sapphire substrates up to 2-inch diameters, with encouraging thickness control for synthesis [31]. In particular, high-quality films for scale-up can be achieved with metal-organic CVD methods [32]. These approaches are also compatible with both atomic layer deposition and with industrial processes, and are thus highly relevant for practical device fabrication [33].

The emphasis of this work is to optimize CVD conditions for MoS_2 synthesis and then evaluate the effects of such parameters on the film's morphology and structure. The intention is that it will facilitate further developments in MoS_2 electronics, sensing and catalytic applications.

Experimental Methods

Synthesis of MoS_2

In Figure 1, the MoS_2 synthesis process is demonstrated. Molybdenum disulfide has been synthesized by the chemical vapor deposition technique. The sources of molyndenum (MoO_3 99,9%, Sigma Aldrich) and sulfur (S 99,9%, Sigma Aldrich) have been placed onto quartz boats in the reaction zone. In the first zone, the sulfur has been placed, the temperature 250 °C was settled. The MoO_3 has been placed into the second zone, and the maximum synthesis temperature of 780 °C was settled for 15 minutes. Argon (Ar 99,99% Ihsan gas) has been used as the transportation gas. A flow of Ar at 220 sccm transports sulfur and MoO_3 vapors to the silicon substrate (Si). The distance between sulfur and MoO_3 was 25 cm, and between MoO_3 and the silicon substrate was 1.5 cm. This configuration provided optimal conditions for the growth of thin MoS_2 on the substrate under the conditions of the used CVD furnace.

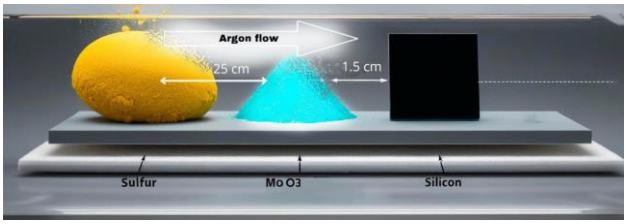

Figure 1 – The process of material synthesis

Investigation of material characteristics

The structural features, such as crystallite shape, size and spatial distribution, were examined for the

samples using scanning electron microscopy (SEM). SEM images were taken using JEOL JSPM-5200 operating at 30 kV accelerating voltage. Energy dispersive X-ray spectroscopy (EDS) measured elemental composition analysis in a JEOL EX-2300 BU detector attached to the SEM system. To keep the spectra consistent with the SEM imaging, EDS spectra were collected under the same conditions.

The layer number and characteristic vibrational mode identification were investigated by Raman spectroscopy. The Raman spectra were obtained using a Jobin-Yvon LabRaman HR800 spectrometer, with monochromatic light of wavelength 632.8 nm.

Results and discussion

The growth conditions are summarized such that the morphological characteristics of the synthesized crystallites are reported in Table 1. Systematic adjustment of the key deposition parameters of CVD synthesis, for example, deposition time and temperature, and the relative positioning of substrate and molybdenum and sulfur sources was done in order to optimize the CVD process. The resulting crystallites were found to have thickness, lateral dimensions and were further confirmed using SEM and Raman spectroscopy.

Figure 2 presents SEM images illustrating the morphology of the synthesized sample. At a magnification of 750× (Figure 2a), the overall surface structure is clearly visible, revealing numerous triangular-shaped crystallites uniformly distributed across the substrate. The lateral dimensions of individual crystallites range from several hundred nanometers to approximately 6 µm, indicating homogeneous growth and a high degree of crystallinity. The observed high nucleation density in certain regions may suggest non-uniform precursor distribution or localized variations in reaction zone parameters such as temperature or reactant concentration [34].

At 9500× magnification (Figure 2b), the fine structure of individual triangular crystallites becomes clearly visible. The well-defined grain boundaries and uniform crystal surfaces observed in the image are indicative of the layered nature of the material and confirm the hexagonal symmetry of the MoS_2 crystal lattice. The formation of triangular and polygonal crystallites can be attributed to anisotropic growth behavior during the CVD process. As Mo and S atoms assemble into hexagonal layers, differences in growth rates along crystallographic directions result in distinct crystal shapes. In particular, when there is an excess of molybdenum, the crystallites tend to adopt a triangular morphology, whereas a more balanced distribution of molybdenum and sulfur leads to more symmetric, nearly hexagonal forms [35].

The results of elemental mapping, which confirm the composition and spatial distribution of elements within the sample structure, are presented in Figure 3. The region selected for analysis is shown in Figure 3a, where triangular and polygonal crystallites are clearly distinguished against the background of the silicon substrate. Figure 3b illustrates the distribution of silicon, which constitutes the underlying substrate. A decrease in silicon signal intensity is observed in the areas covered by MoS_2 crystallites, indicating uniform deposition of the material across the substrate surface.

Figure 3c shows the distribution of molybdenum. The high Mo signal intensity is localized in the regions where MoS_2 crystallites have formed, confirming the presence of molybdenum disulfide. The gradient in signal intensity suggests variations in layer thickness, which may be attributed to growth kinetics under conditions of limited precursor availability. The lower right panel displays the distribution of sulfur, which, in contrast to molybdenum, appears more diffuse. This may indicate compositional variations across the sample or the presence of amorphous sulfur species deposited onto the substrate.

The formation of well-ordered crystallites is governed by a combination of factors, including crystallographic growth anisotropy, thermodynamic constraints, and nucleation mechanisms [34]. The hexagonal structure of MoS_2 promotes preferential growth along low-energy crystal planes, resulting in the formation of triangular and hexagonal platelets. Synthesis temperature plays a particularly critical role; under optimal conditions, a balance between nucleation and crystal growth is achieved, enabling the formation of uniform, highly crystalline structures. The gradient elemental distributions observed in the EDS maps further support the kinetic nature of the deposition process and reflect local compositional fluctuations during film formation.

Table 1 – Morphological characteristics of MoS_2 crystallites under various CVD synthesis conditions

Synthesis Temperature (°C)	Deposition Time (min)	Crystallite Size (μm) and Thickness (nm)	Substrate Position Relative to Mo Source	Comments
620	10	~1–7 μm, ~200–300 nm	Distance between sulfur and MoO_3 - 25 cm between MoO_3 and substrate - 5 cm	Uniform circular structures formed on the substrate surface, with sulfur-rich composition
700	10	~4–7 μm, –	Distance between sulfur and MoO_3 - 30 cm between MoO_3 and substrate - 5 cm	Needle-like structures with molybdenum enrichment
750	10	~2–5 μm, ~0.7 nm	Distance between sulfur and MoO_3 - 30 cm, between MoO_3 and substrate - 1.5 cm	Triangular structures formed with partially developed edges
780	15	~2–6 μm, ~0.7 nm	Distance between sulfur and MoO_3- 25 cm, between MoO_3 and substrate - 1.5 cm	Well-defined triangular crystallites with sharp edges

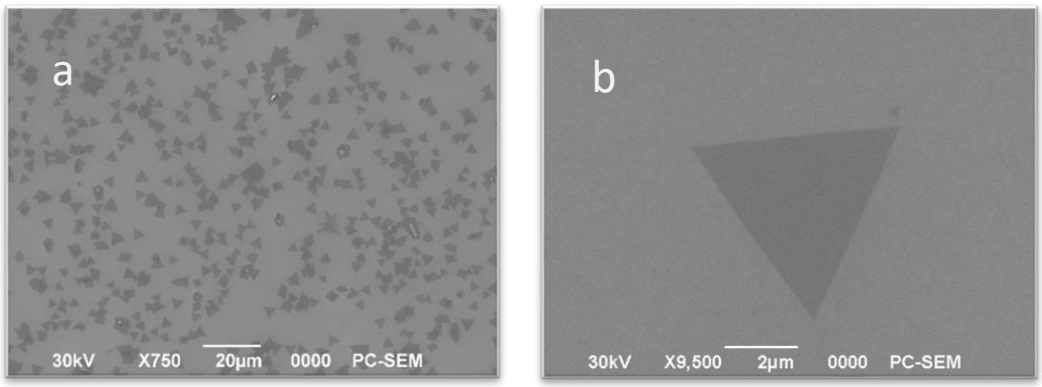

Figure 2 – SEM images of the surface morphology of the MoS_2 sample: (a) 750× magnification; (b) 9500× magnification

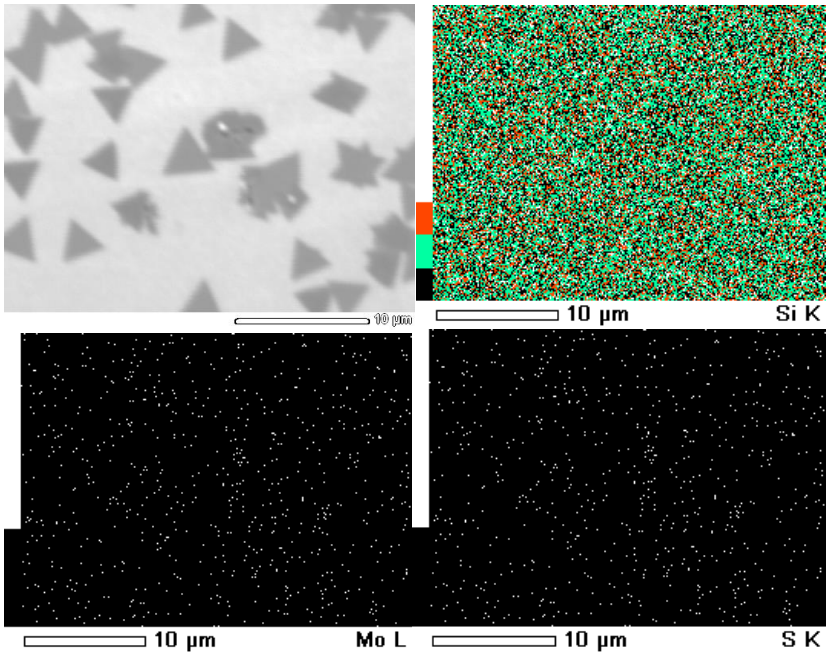

Figure 3 – Elemental mapping of the MoS_2 sample obtained by energy-dispersive X-ray spectroscopy (EDS)

The crystal structure was analyzed using Raman spectroscopy. The spectra were taken at normal temperature with a single-colour light beam at 632.8 nm wavelength. The measurements were done using a 100× objective lens, which focused a laser beam of 1 µm diameter. A single crystallite from the MoS_2 sample provided the spectra presented in Figure 4. The optical image of this sample is shown in the upper-left corner of the spectra. The spectra show two sharp peaks at ~384 cm^{-1} and ~405 $cm^{-1,}$ which are characteristic vibrational modes of MoS_2 known as E_{2g}^1 and A_{1g}. These vibration modes are located at a distance of Δ ≈ 20.9 $cm^{-1,}$ which shows clear signs of a single layer of MoS_2. The Raman spectra exhibit sharp and intense E_{2g}^1 and A_{1g} peaks, indicative of high crystallinity and structural order in the monolayer MoS_2 [36]. A well-defined structure with a high specific surface area facilitates efficient charge carrier separation and offers numerous active sites for hydrogen evolution reactions [37]. The SEM images show clear edge structures and a uniform pattern, which shows that this sample has many surface locations that react efficiently. An effective resistive gas sensor works through specific edge locations that preferentially take gas molecules and alter electrical conductivity [38].

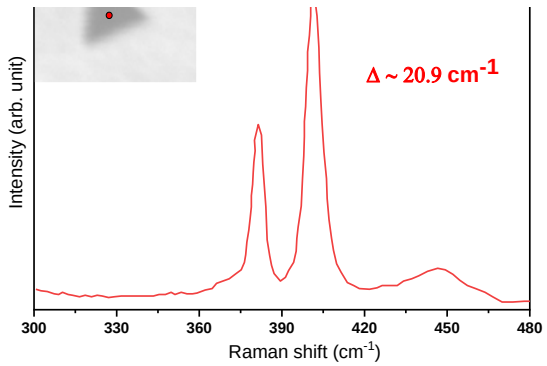

Figure 4 – Raman spectrum of the MoS_2 sample

The two-phonon scattering process at 450 cm^{-1} shows up as a broad peak in the spectrum because this band appears in layered transition metal dichalcogenides [35]. The small peak ratio and narrow lineshapes of E_{2g}^1 and A_{1g} prove the high-quality MoS_2 monolayer formation.

Conclusion

The combined results of SEM imaging and elemental mapping indicate that the synthesized MoS_2 exhibits high crystallinity, well-defined structure, and a characteristic morphology shaped by growth dynamics under CVD conditions. Elemental distribution analysis confirms the uniform incorporation of molybdenum and sulfur within the crystalline domains, along with some compositional variation at grain boundaries. Raman spectroscopy further verifies that the obtained sample corresponds to a monolayer of MoS_2. The interpeak distance of Δ ≈ 20.9 cm^{-1} between the E_{2g}^1 and A_{1g} modes is consistent with high-quality monolayer formation. SEM analysis corroborates the uniform spatial distribution of crystallites and their distinct hexagonal morphology. These findings provide a solid basis for further optimization of MoS_2 synthesis parameters aimed at tailoring morphological characteristics, which is particularly relevant for applications in electronic and optoelectronic devices.

Conflicts of interest. The authors declare no conflict of interest.

CRediT author statement: **Ye.Otunchi**: Methodology; **A. Umirzakov**: Formal analysis; **E. Dmitriyeva** and **A. Shongalova:** Writing-original draft; **A. Kemelbekova**: Writing review. All authors have read and agreed to the published version of the manuscript.

Acknowledgements. This work was financially supported by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan under Grant No. BR21881954.

Cite this article as: Otunchi Ye, Umirzakov A, Dmitriyeva E, Shongalova A, Kemelbekova A. Morphological and Crystallographic Investigation of CVD-Grown MoS_2. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2026; 339(4):30-37. https://doi.org/10.31643/2026/6445.38

CVD әдісімен алынған MoS_2-нің морфологиясын және кристаллдық тор құрылымын зерттеу

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Мақала келді: *14 ақпан 2025*
Сараптамадан өтті: *8 сәуір 2025*
Қабылданды: *9 маусым 2025*

ТҮЙІНДЕМЕ

Мақалада бу фазасынан химиялық тұндыру (CVD) әдісімен алынған MoS_2 негізіндегі перспективалы материалдың құрылымдық сипаттамалары зерттелген. Қажетті құрылымды алу үшін синтез процесін оңтайландыру нәтижелері де ұсынылған. CVD әдісімен MoS_2 кристалдарын синтездеу үшін оңтайлы параметр күкірттенудің максималды температурасы 780 °C, ұсталу уақыты шамамен 15 минут, күкірт көзі аймағының қыздыру температурасы 250 °C, күкірт пен молибден көздерінің арасындағы қашықтық 25 см, ал молибден көзі мен төсеніш арасындағы қашықтық 1,5 см болды. Алынған үлгілердің морфологиясы мен элементтік құрамы сканерлеуші электронды микроскопия (СЭМ) және энергия-дисперсиялық рентген спектроскопиясы (ЭДС) әдістері арқылы зерттелді. СЭМ нәтижелері бойынша MoS_2 кристалдары үшбұрышты пішінде түзілген және төсеніш бетінде біркелкі таралған. Кристалдардың ең үлкен өлшемі 6 микронға дейін жетеді. ЭДС-картографиялау нәтижесінде молибден мен күкірттің кристал құрылымында біртекті таралуы анықталды, тек түйіршіктер шекараларында аздаған құрам ауытқулары байқалды. Үлгінің сапасы мен қабат саны Раман спектроскопиясы арқылы зерттелді. Спектрде MoS_2 наноқабатының екі сипаттамалық шыңы (E_{2g}^1 және A_{1g} тербеліс режимдерінде) тіркелді, шыңдардың пішіні өткір формада, олар ≈20.9 $см^{-1}$ қашықтықта орналасқан, бұл алынған кристалдардың жоғары құрылымдық сапасын көрсетеді. Алынған нәтижелер таңдалған тәсілдің тиімділігін және жұмыс нәтижелерінің екі өлшемді материалдар технологиясын дамытудағы маңыздылығын дәлелдейді.

Түйін сөздер: молибден дисульфиді, CVD синтезі, 2d материалдар, Раман спектроскопиясы, морфология.

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Морфологическое и кристаллографическое исследование MoS_2 выращенных CVD-методом

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Поступила: *14 февраля 2025*
Рецензирование: *8 апреля 2025*
Принята в печать: *9 июня 2025*

АННОТАЦИЯ

В данной статье представлено исследование структурных характеристик перспективного материала на основе MoS_2, полученного методом химического осаждения из паровой фазы (CVD). Также представлена оптимизация процесса синтеза для получения желаемой структуры. Оптимальным параметром синтеза методом CVD MoS_2 кристаллов было выявлено максимальная температура сульфуризации 780 °C с выдержкой около 15 минут, температура нагрева зоны источника серы 250 °C, расстояние между источниками серы и молибдена 25 см, а также расстояние между источником молибдена и подложки составляло

	1,5 см. Морфология и элементный состав полученных образцов были изучены с помощью сканирующей электронной микроскопии (СЭМ) с энергодисперсионным рентгеновским спектроскопией (ЭДС). С помощью СЭМ было выявлено, что кристаллы MoS_2 формированы треугольной формы и равномерно распределены по поверхности подложки. Максимальные размеры кристаллитов достигают 6 мкм. ЭДС-картирование кристаллитов подтвердило однородное распределение молибдена и серы в структуре, выявив лишь незначительные вариации состава на границах зерен. Качество, количество слоя образца были изучены с помощью Рамана спектроскопии. Результаты показали два характерных пика (vibrational modes E_{2g}^1 and A_{1g}) наноразмерных MoS_2. Пики имеют острую форму и расположены на расстоянии ≈20,9 $см^{-1}$, что может свидетельствовать о высоком качестве кристаллической структуры полученных кристаллитов. Полученные результаты подчёркивают эффективность выбранного подхода и значимость работы для развития технологий 2D-материалов.
	Ключевые слова: дисульфид молибдена, CVD-синтез, двумерные материалы, Рамановская спектроскопия, морфология.
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