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Molybdenum Disulfide Chemical Vapor Deposition Growth and Raman Characterization

by Mitchell Hung, Kai Palam, Madan Dubey, and Charles Rong

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14. ABSTRACT Molybdenum disulfide (MoS ₂) is a transition metal dichalcogenide (TMD) that has garnered attention as a potential alternative to silicon-based electronics. MoS ₂ exhibits a direct band gap and is atomically flat and thin, making it ideal for use in transistors and optoelectronics. The ability for MoS ₂ to be grown in monolayers due to its layered structure—molybdenum sandwiched by sulfur—gives the material a distinct advantage in precise growth. However, the growth of monolayer MoS ₂ depends on a range of growth factors, including the choice and volume of growth promoters. We analyzed the effects of varying volumes of potassium bromide (KBr), the chosen growth promoter, on the shapes and properties of MoS ₂ triangles. Using Raman spectroscopy, we observed that different KBr volumes led to changes in the spectral signatures and the formation of MoS ₂ structures. Furthermore, we found via Raman peak and waveform analysis that lower volumes of KBr resulted in more consistent, repeatable results. This research supports the development of an automatic classification system for MoS ₂ layers.					
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1. Introduction

Silicon has been prominent in our lives through its presence in the semiconductor industry and the electronics we use nearly every day. Over 70 years of development, researchers have improved materials quality, processing technique, device fabrication and manufacturing capability, leading to consistent progress with transistor scaling and circuit design. However, progress is stagnating after reaching the atomic scale, as silicon faces difficulties regarding its material properties. Issues such as subthreshold leakage and hot carrier injection, while being actively researched, have still slowed the pace of Moore's law. A new, alternative material with superior electronic and optical properties could overcome these limitations.

Transition metal dichalcogenides (TMDs) have emerged as potential materials to complement silicon in electronic and optoelectronic applications. In particular, molybdenum disulfide (MoS_2) has attracted attention due to its semiconducting behavior with a direct bandgap of approximately 1.8 eV in the monolayer form, high on/off ratios, and reasonable carrier mobility.^{1,2} This electronic performance, coupled with its flexibility, makes it suitable for the development of novel electronic devices.

Yet, despite the advantages of MoS_2 , a well-controlled synthesis of high-quality MoS_2 has been a challenge. Chemical vapor deposition (CVD), the most promising candidate for creating large-area monolayers due to its controllability, still suffers from nonuniform growth leading to incomplete coverage of substrate. However, recent studies show that alkali halide salts, such as potassium chloride (KCl) can serve as growth promoters that reduce nucleation energy and enhance lateral domain growth.³ Here, building on this research, we investigate the use of potassium bromide (KBr) as a growth promoter for the CVD synthesis of MoS_2 .

This technical report presents an analysis of MoS_2 growth using KBr under varying conditions with the goal of improving uniformity, coverage, and overall film quality. The results contribute to the ongoing effort to identify optimal growth strategies for 2D semiconductors and to expand the range of materials available for future electronic and optoelectronic technologies.

2. Background

We briefly define and discuss relevant concepts in the growth of MoS_2 literature that are relevant to this research and outline related work in these areas and the existing scientific gaps that this research addresses.

2.1 MoS₂

MoS₂ is a layered TMD with the chemical formula MX₂, where M = molybdenum and X = sulfur. Its crystal structure is composed of a layer of molybdenum atoms sandwiched between two layers of sulfur atoms in a trigonal prismatic structure⁴ as a “plane” or layer. These layers are bonded strongly by covalent bonds within the plane/layer, and weakly by van der Waals forces between planes/layers, allowing MoS₂ to be exfoliated into atomically thin layers and grown with methods such as CVD. One of the most important features of MoS₂ is the thickness-dependent nature of its electronic band structure. Bulk MoS₂ possesses an indirect bandgap of approximately 1.2 eV, whereas a single monolayer possesses a direct bandgap of approximately 1.8 eV. This direct gap enables more efficient light absorption and emission, highlighting monolayer MoS₂ with a growing potential for optoelectronic devices such as photodetectors, LEDs, and solar cells.^{4,5}

In terms of electronics, as previously discussed, monolayer MoS₂ shows high on/off current ratios (up to 10⁸) and better than average carrier mobilities, sometimes reaching approximately 500 cm²/V·s under optimized conditions.⁶ Its ability to overcome short-channel effects, coupled with its flexibility and stability, extends its utility toward flexible and wearable electronics.^{7–9}

Despite these advantages, challenges remain in producing MoS₂ films with uniform thickness, controlled orientation, and low defect density at wafer scale. Various strategies including growth promoters, halide-assisted growth, and environmental variation are being explored to address these limitations. As a result, MoS₂ research currently sits at the intersection of materials science and applied device engineering, with progress in synthesis dictating the feasibility of its technological adoption.

2.2 CVD

CVD is a widely adopted technique used to grow high-quality thin films by delivering precursors that react at high temperatures to form solid materials on a substrate. With the growth of MoS₂, CVD stands out for its scalability, uniformity, and compatibility with semiconductor manufacturing.¹⁰

For MoS₂ synthesis, the most common strategy involves allowing vaporized pure sulfur to react with molybdenum oxide (MoO₃) or halide precursors (e.g., MoCl₅) at temperatures typically in the range of 700–1000 °C.¹¹ At higher temperatures, sulfur anneals lead to more symmetrical characteristic peaks.^{11,12}

Scalability and uniform coverage remain ongoing challenges. Recent research has addressed this by regulating oxygen dosing to merge MoS₂ domains into wafer-

scale films with low defect densities and enhanced optoelectronic performance, enabling devices like centimeter-scale photodetector arrays.⁷

A more practical insight comes from a 2017 study¹³ demonstrating an upstream deposition approach in a multizone CVD setup. By controlling the flow and location of Mo and S precursors, researchers achieved high nucleation density, uniform monolayers, and improved charge capture capabilities—a significant step toward reliable device-grade MoS₂ growth.¹³

2.3 Growth Promoters

Growth promoters significantly enhance MoS₂ synthesis via CVD by improving nucleation control, lateral domain expansion, film uniformity, and even electrical properties.

Organic seeding promoters such as perylene-based compounds (for example, perylene-3,4,9,10-tetracarboxylic dianhydride [PTCDA] or perylene-3,4,9,10-tetracarboxylic acid tetra-potassium salt [PTAS]) have been adopted for improving monolayer coverage and grain size. Using a promoter-coated substrate placed downstream allows for a gradient of promoter diffusion upstream. Regions closer to the promoter show nearly continuous polycrystalline monolayers with bilayer terraces, while farther areas display isolated triangular monolayer domains alongside multilayers.¹⁴

Inorganic promoters, especially alkali salts like KCl, NaCl, KI, and molten salt mixtures, act to facilitate lateral growth and enhance the size of triangle MoS₂. Studies indicate that adding alkali-based salts significantly increases lateral expansion and substrate coverage, yielding high-quality bilayer films, with potassium salts often outperforming others in uniformity and coverage.^{15–17}

2.4 Our Goal

MoS₂ is in the middle of a research boom, as it continues to emerge as one of the most promising 2D materials for applications ranging from flexible transistors and neuromorphic computing to next-generation processors and high-capacity batteries.^{3,6–9} Yet, the field is held back by a “scaling bottleneck.” Although the fundamental optical and electronic properties of MoS₂ have been discovered, producing wafer-scale films with controlled thickness, low defect density, and high uniformity remains difficult. This gap between knowledge and the ability of scalable synthesis sustains great interest in discovering new growth strategies. Our work on KBr-assisted CVD seeks to address these difficulties, testing whether KBr can promote more triangular domain growth and monolayer coverage. By

combining synthesis with Raman-based analysis¹⁸ of peak separation, full width at half maximum (FWHM), and various other characteristics, we aim to develop both a deeper understanding of structure–property relationships and practical workflows for optimizing MoS₂ growth at larger scales.^{19–21}

While optical image of MoS₂ grown on a wafer only provides information of wafer coverage and morphology of MoS₂, Raman spectroscopy will provide further structural information related to number of layers and defect level, which is more helpful to understanding the formation of MoS₂, and optimize the growth conditions. Raman peak position, relative intensity, linewidth, and peak separation can all possibly correlate to the crystal structure and materials quality. These spectral fingerprints can guide the optimization of MoS₂ for device-relevant properties such as charge transport in semiconductors and ion storage in batteries.

2.5 Specific Aims

The specific aims of this work is to do the following:

- 1) Measure and characterize the Raman spectra of inelastic light scattering (Raman peak) properties and their relation to 2D image characteristics
- 2) Develop methodology to characterize unexplained residuals of Raman spectra after peak analysis
- 3) Develop workflow to accelerate dense sampling of 2D surfaces

3. Methodology

MoS₂ films were synthesized via CVD using MoO₃ (Sigma-Aldrich, ≥99.5%), elemental sulfur (sulfur flakes, Sigma-Aldrich, ≥99.99%), and KBr (Sigma-Aldrich, 99+%), as the precursor materials. A solution of MoO₃ and KBr was prepared and spin-coated onto silicon wafers (with 100 nm of SiO₂ on Si), which had been precleaned in acetone. Spin coating was performed at 2500 rpm using a Model PWM32 spin coater, ensuring uniform deposition of the precursor layer.

The coated wafers were then transferred into the reaction chamber of a Lindberg/Blue M tube furnace. Elemental sulfur was placed upstream in the argon carrier gas flow to provide the sulfur vapor source. The furnace was heated to 720 °C over a period of 15 min, maintained at this temperature for 5 min, and subsequently cooled rapidly to room temperature. During the entire process, argon gas flowed at a rate of 30 SCCM to transport the vaporized sulfur into the growth region, where it reacted with the MoO₃/KBr-coated substrate to form MoS₂.

The procedure for making the MoO₃/KBr solution is described below. First, 100 mg of MoO₃ was added to 20-mL NH₃•H₂O and the mixture was sonicated for 5 min, until all the solids dissolved in the solution. To this clear solution, 60 mL of deionized water was added to create Solution A. Second, 720-mg KBr was dissolved in 20 mL of deionized water as Solution B. From Solution A and Solution B, more solutions were made to have different concentrations of KBr with same concentration of MoO₃ as we varied the amount of KBr in the MoO₃–KBr precursor solution. Specifically, three solutions were made by adding Solution B in volumes of 2.0, 1.0, and 0.5 mL to each of the 20-mL Solution A. In addition, a separate trial was conducted with 0.5-mL KBr, in which the growth temperature of 720 °C was maintained for 10 min rather than 5 min to double the reaction time to assess the effects of extended high-temperature growth time.

Characterization of the resulting MoS₂ films was performed using a WITec alpha300 Access Raman Microscope (WITec Control Software, Version 1.60). Spectra were collected using a 532-nm excitation laser. First, measurements were acquired in acquisition of single-spectrum mode, with an integration time of 2.0 s per spectrum and 20 accumulation cycles, to get sufficient signal-to-noise ratio. Toward the end of the research process, measurements were also acquired in large-area scan mode for a mapping purpose. The large-area scan mode produced a collection of Raman measurements at each location defined by the point and line parameters in the spectrometer for the selected scanned area. Each Raman measurement produced a vibration absorption intensity of the E_{2g} mode around 380 cm⁻¹ and of the A_{1g} mode around 400 cm⁻¹. The Raman shift was calibrated relative to the 550 nm reference setting.

Acquired data of the large-area scans were subsequently analyzed via custom Python scripts to process the information of Raman characteristics related to structural and morphological MoS₂ distribution of mono and multiple layers, providing insight into layer thickness and crystallinity of the MoS₂ films.

4. Results

4.1 Data Analysis Overview

The Raman spectra were processed using Python-based analysis to determine the precise locations of characteristic peak positions. From these values, the peak separations and other relevant spectral parameters were calculated, subsequently mapped into color and shape representations, and visualized as graphs. The Python code and other programming-related supplementals can be found in the Appendix.

4.2 Large-Area Scans

In addition to single spectrum acquisition function, the WITec Control software provides a large-area scan feature, enabling automated Raman spectroscopy scanning across a region of interest. In this mode, the selected area is subdivided into a grid, and spectra are collected at each point to generate Raman maps. The spatial resolution of these scans can be adjusted by varying the step size, thus balancing scanning time and map resolution to meet each researcher's requirements.

Figure 1 demonstrates scans of the same $100 \times 100 \mu\text{m}^2$ region acquired at different resolutions. Higher-resolution scans provide finer spatial details at the cost of longer scanning times, whereas lower-resolution scans enable faster sampling with the caveat of reduced precision. This functionality allows comparison of growth uniformity, domain distribution, and layer thickness across large regions of the MoS₂ samples without manual human labor after setup.

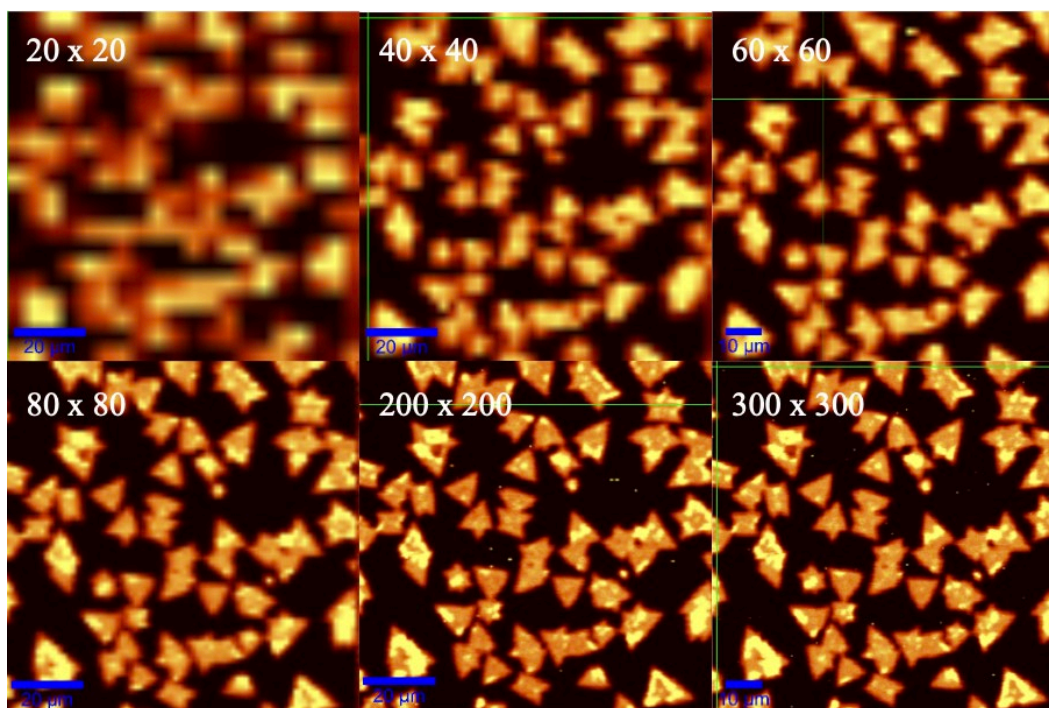


Figure 1. Comparison of various resolutions. With the scanned size of $100 \times 100 \mu\text{m}$, the point per line and lines per image varies from 20×20 at low resolution, up to 300×300 at high resolution.

Table 1 summarizes the Raman large-area scan parameters.

Table 1. Large-area scan parameters. Data gathered under 2-s integration time with 20 integration cycles.

No. of measurements in the X axis	No. of measurements in the Y axis	Total no. of measurements	Total time (h)
20	20	400	0.25
40	40	1,600	1.00
60	60	3,600	2.16
80	80	6,400	3.84
200	200	40,000	24.00
300	300	90,000	54.00

4.3 Effects of KBr Concentration

By analyzing the peak properties of MoS₂ triangles across different growth conditions, we observed that samples synthesized with the precursor solution of 0.5 mL of Solution B exhibited peak separations clustered more tightly around 21.12 cm⁻¹. This narrower distribution suggests that growth under these conditions leads to more consistent and reproducible results, highlighting the precursor solution made of 0.5 mL of Solution B as a promising parameter for synthesis.

Raman spectra were further analyzed by identifying the characteristic E_{1g} and A_{1g} vibrational modes of MoS₂ and calculating the separation between their peak positions. Whereas in past work peaks were identified manually, here we accelerated this analysis via combination of large-area scan analysis and custom Python code. We then created a colorized map of the peak separations (Figure 2) across selected triangular domains of the MoS₂ films.

Based on the peak separation map in Figure 2a, regions where triangular domains overlap exhibited wider peak separations, whereas edge regions had narrower separations. This trend is consistent with established Raman behavior in MoS₂, where monolayers typically show peak separations near 19–20 cm⁻¹ and multilayers approach values of 23–25 cm⁻¹.

These results suggest that the overlapped triangular regions correspond to thicker, multilayer MoS₂, whereas the edges are more representative of monolayer domains. The variation across the mapped area indicates that the CVD growth produced nonuniform films, with localized thickness changes arising from overlapping growth fronts. Such structural heterogeneity is significant, as the electronic and optical properties of MoS₂ are strongly thickness-dependent: monolayers exhibit a direct bandgap favorable for optoelectronics, while multilayers transition to an

indirect bandgap with diminished performance in those applications. Consequently, the observed peak separation distribution highlights both the challenges and opportunities in optimizing KBr-assisted CVD growth, emphasizing the importance of large-area Raman mapping for uniformity and process refinement.

Additionally, we developed a method to extract the individual Raman spectra from the large-area scan dataset. This represents an important step toward the broader goal of automating the identification of MoS₂ triangular-shaped domains and quantifying their properties related to structure. By isolating individual spectra, we can directly evaluate peak separation values at specific locations across the scanned region. We then used these values to generate a reconstructed map of the large-area scan, where each point is represented according to its characteristics observed by Raman spectroscopy. In particular, the analyzed characteristics include the E_{2g} peak, the A_{1g} peak, as well as ratios such as an intensity ratio and an FWHM ratio between the E_{2g} and A_{1g} peaks.

Peak locations were determined by taking the maximum intensity value within a predetermined common range for those mode peaks—380–400 and 400–420, respectively—and finding the Raman shift corresponding to those maximum values. Intensity ratio took the ratio between the two maximum intensity values, with the A_{1g} peak as the dividend. FWHM was calculated by taking the maximum value of a peak, finding the minimum peak intensity within the aforementioned range as a baseline, and finding which Raman shifts lined up with the midpoint between maximum and baseline. FWHM Ratio took the ratio between the FWHMs of each peak, also with the A_{1g} peak as the dividend. When peak intensities in each range varied by less than 20 a.u., the data were thrown out (not applicable).

Figure 2 demonstrates the potential value of some characteristics over others. In particular, when observing Figures 2a and 2d, they clearly have stronger distinction between certain areas of the MoS₂ flakes, potentially signifying the ability to detect monolayer versus bilayer triangular regions. On the other hand, Figures 2b and 2c suggest a lowered sensitivity to layer thickness, potentially making them suboptimal for thickness classification. Figure 2e suggests mixed results, with some level of thickness separation but at a lesser rate than Figures 2a and 2d.

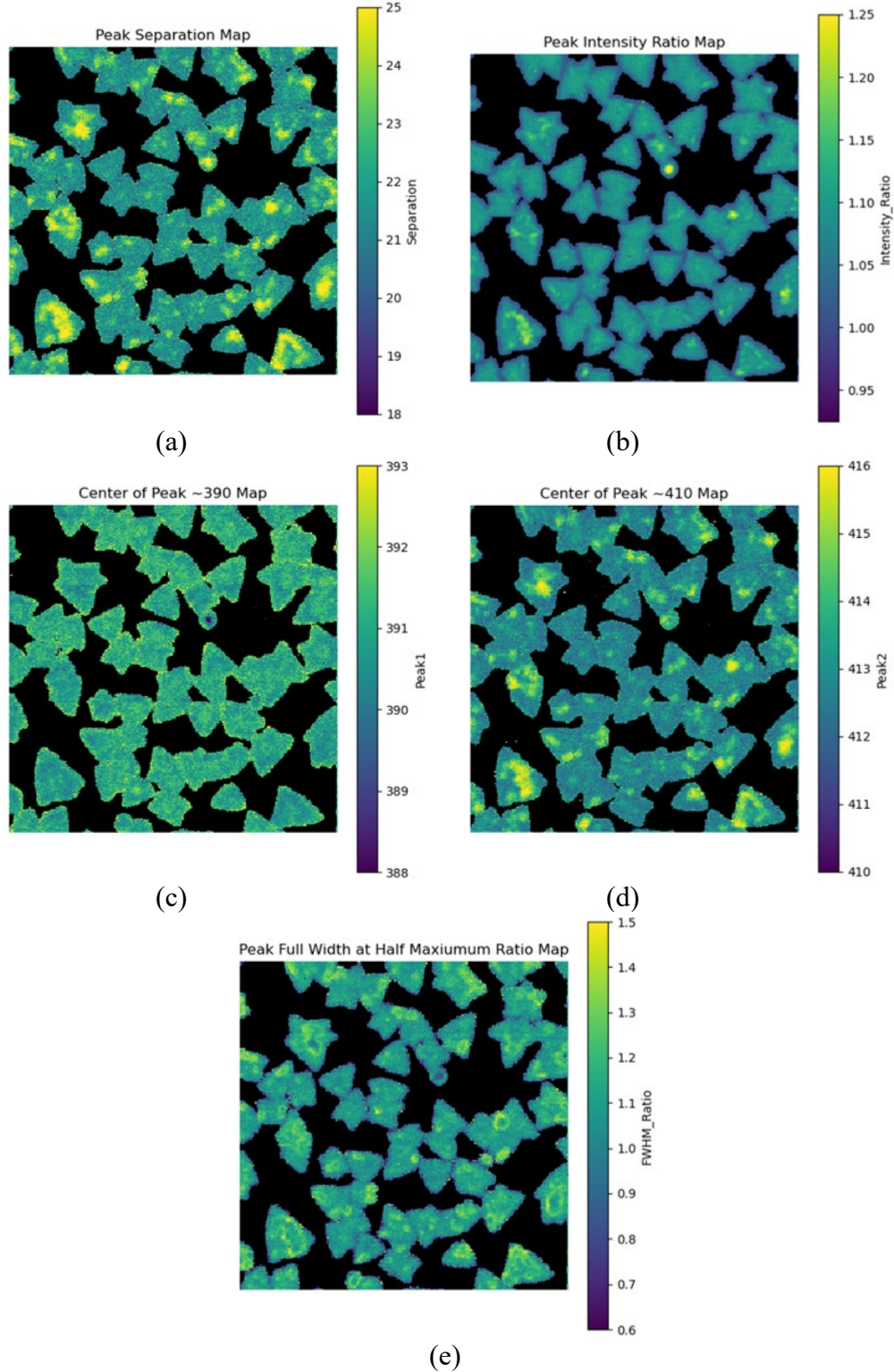


Figure 2. Reconstructed large-area (a) scan with color based on separation between the A_{1g} and E_{1g} peaks, (b) scan with color based on intensity ratio between the A_{1g} and E_{1g} peaks, (c) scan with color based on the Raman shift of the A_{1g} peak, (d) scan with color based on the Raman shift of the E_{1g} peak, and (e) scan with color based on the FWHM ratio between the A_{1g} and E_{1g} peaks.

5. Discussion

Through the development of Raman large-area scan analysis, we find that reduced promoter concentrations may enhance film uniformity and that overlapped triangles form thicker, multilayer MoS₂, worsening film quality. Such structural variation is significant, as the electronic and optical properties of MoS₂ are highly thickness-dependent: monolayers exhibit a direct bandgap favorable for optoelectronics, while multilayers transition to an indirect bandgap with diminished performance in those applications. Thus, the observed peak separation distribution highlights both the challenges and opportunities in optimizing KBr-assisted CVD growth, emphasizing the importance of large-area Raman mapping for evaluating uniformity and guiding process refinement.

Analysis of MoS₂ growth via Raman spectra peak separation is a well-established method, and relatively simple to measure. Yet, the possibility of a more subtle pattern present in the Raman signals cannot be excluded. Additionally, this research does not address the role of residuals in MoS₂ Raman spectra not explained by simple measures of peak height and peak separation, leaving open the possibility for future analysis that could yield new insights. Moreover, during the research process, samples of MoS₂ were scanned using photoluminescence (PL) spectroscopy. These PL datasets, while not analyzed in this report, could present a valuable resource for future investigations of MoS₂ properties.

The findings of this study extend beyond MoS₂ growth with KBr as promotor, implying broader inclusions for the synthesis of other 2D TMDs. The methodology developed, combining large-area scan mapping of Raman measurements with data processing and analysis enabled by Python programming, can be generalized to investigate nucleation, uniformity, and defect density in a variety of other layered materials such as WS₂, MoSe₂, and WSe₂. Furthermore, while our experiments were conducted on relatively small silicon substrates, this approach is directly scalable to larger wafers, which are common in the industry.

6. Conclusion

This work investigated KBr as a growth promotor in the CVD synthesis of MoS₂ and developed a new method of analyzing TMD samples. While challenges remain for fully characterizing the material's properties, the methodologies developed here provide a platform for enhanced characterization capability to the need for optimized 2D semiconductor growth. Large area Raman scan and Python programming to process structural information related quality of MoS₂ material and the atomic layer(s) distribution can be extended to other 2D material systems.

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Appendix. Python Source Code Files for Data Treatment

This appendix appears as a pdf attached to the report.

<https://github.com/Ghostyaq/Molybdenum-disulfide-CVD-Growth-with-Potassium-Bromide-Precursor.git>

List of Symbols, Abbreviations, and Acronyms

2D	Two-Dimensional
CVD	Chemical Vapor Deposition
FWHM	Full Width Half Max
KBr	Potassium Bromide
KCl	Potassium Chloride
LED	Light Emitting Diode
MoO ₃	Molybdenum Oxide
MoS ₂	Molybdenum Disulfide
PL	Photoluminescence
PTAS	Perylene-3,4,9,10-Tetracarboxylic Acid Tetra-Potassium Salt
PTCDA	Perylene-3,4,9,10-Tetracarboxylic Dianhydride
SCCM	Standard Cubic Centimeters per Minute
Si	Silicon
TMD	Transition Metal Dichalcogenide

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