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REGULAR ARTICLE

Clustering-Based Growth Analysis of 2D Transition Metal Thin Films on Graphene Substrates via Molecular Beam Epitaxy

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Metal dichalcogenides are a kind of chemical substance that consists of a metal atom paired with chalcogen elements such as selenium and sulphur. These materials have distinctive electrical and optical characteristics, making them fascinating for a variety of applications, including electronics and optoelectronics. Growth examination of metal dichalcogenide thin films entails analyzing their controlled deposition and crystallization. Understanding growth processes, substrate interactions and controlling parameters like as temperature and precursor concentration are critical for producing high-quality films with the appropriate characteristics, establishing the way for developments in nanotechnology and device manufacturing. Throughout this research, we employed the Machine learning (ML) enabled Reflection High-Energy Electron Diffraction (RHEED) analytical approach to examine the development of two-dimensional (2D thin layers of dichalcogenides ($ReSe_2$) made of transition metals on graphene substrates using Molecular Beam Epitaxy (MBE). Independent Component Analysis (ICA) and the Fuzzy C-Means approach were implemented to determine different patterns and represent the pattern growths. To decrease the original dataset's dimensionality, we employed 20 Independent Components (ICs) and each RHEED image was distributed to the closest centroid, which resulted in the dataset being clustered using Fuzzy C-Means.

Keywords: Growth Analysis, 2D Transition Metal Dichalcogenide, Graphene Substrates, Molecular Beam Epitaxy (MBE), Fuzzy C-Means.

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1. INTRODUCTION

In the fields of materials science and nanotechnology, two-dimensional (2D) material exploration has garnered a great deal of interest lately. According to their special mechanical, optical and electrical characteristics, Transition Metal Dichalcogenides (TMDs) have grown to be among the most promising solutions for a variety of uses [1]. It is essential to comprehend these materials' growth processes to modify their properties to satisfy certain application needs.

Graphene is a perfect substrate for improving the performance of 2D materials because of its remarkable mechanical strength and electrical conductivity [2]. The systematic study of the growth dynamics of TMD thin films on graphene is made possible by the precise and regulated thin film deposition method known as molecular beam epitaxy [3]. Transition metal dichalcogenides (TMDs) in 2D on graphene substrates are an intriguing field of study with broad implications for numerous industrial applications [4].

TMDs are a family of materials distinguished by a structure akin to a sandwich, whereby chalcogen atoms (sulfur, selenium, or tellurium) are sandwiched between a layer of transition metal (such as tungsten or molybdenum). Because of their special structure, which results in unique electronic and optical properties, they are appealing for use in optoelectronics and other fields in addition to electronics [5].

This work intends to use molecular beam epitaxy (MBE) to investigate the formation of two-dimensional (2D) thin layers of dichalcogenides ($ReSe_2$) made of transition metals on graphene substrates using ML enabled RHEED.

The following divisions are found in the remaining portion of this study: part 2 contains a related work; part 3 has the methodology; part 4 discusses the analysis of

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the research and part 5 contains the research's conclusion.

2. RELATED WORK

The paper [6] examined the basic principles of Van der Waals (VDW)-bonded crystal epitaxial development that was particular to Transition Metal Dichalcogenide (TMD) films. Initially, sources and techniques for vapor phase deposition were presented together with the structural and electrical properties of TMD crystals. TMD epitaxy-specific concerns, such as substrate characteristics, bonding and orientation between the film as well as substrate were reviewed critically.

The study [7] demonstrated the use of molecular beam epitaxy to generate Hafnium Diselenide (HfSe2) thin films. They were able to show multilayer, crystalline development on various 2D substrated like Highly Ordered Pyrolytic Graphite (HOPG) and Molybdenum Disulfide (MoS2) without misfit dislocations because of the reduced growth conditions. Following an atomically sharp interface with the substrate, flat, two-dimensional layers with octahedral coordination were seen in the $HfSe_2$ thin films.

The paper [8] looked at the processes used to create TMDCs. It discussed their characteristics and paid special attention to their topological, superconductive and charge-density-wave phases. In addition to methods for improving the mobility of charge carriers' high-frequency functioning and the use of strain science to modify their characteristics, the application of TMCDs in nano-electronic devices was examined.

The article [9] concentrated on the latest development in comprehending the properties and actions of several applications for 2D stacked graphene/TMD hybrid structures. In that context, they first went over the properties of electron transport at the interface between TMD heterostructures and graphene. After that, the tunable characteristics of those hetero-structures were assessed considered their various architectural designs.

The study [10] focused on many techniques to create bi-layer VDW materials has been devised. The characteristics and production of TMDs and Bilayer Graphene (BLG) has advanced, they highlighted how the composition, stacked arrangements and twisted angles of BLG and TMDs could affect their fascinating features. Remarkably, BLG and TMDs with huge domain size, good quality and strong interlayer coupling were grown on a massive scale using Chemical Vapor Deposition (CVD).

The study [11] presented surface Plasmon Resonance (SPR) simulations of two-dimensional materials, notably graphene, graphene oxide, molybdenum disulfide and tungsten diselenide, in monolayer and multilayer architectures. They looked into the SPR biosensors' Deoxyadenosine (DA) and sensitivity to hybrid structures. According to the acquired results, compared to traditional structures, the sensitivity of the SPR could be increased by up to five times by the usage of 2D material.

The paper [12] provided a planar epitaxial method for the creation of entangled 2D semiconductor monolayer utilized gas-confined chemical vapor deposition, wherein site-selective development of Gr–WS_2 (Tungsten disulfide)–Gr and Gr–WSe_2(Tungsten Diselenide)–Gr hybrid structures was guided by patterned Graphene (Gr). The Gr/2D interface for semiconductors showed a transparent contact with virtually perfect securing factors of 0.95 and 0.92 for WSe_2, the *p*-channel along with WS_2, the *n*-channel.

The study [13] investigated a density functional theory of the electrical, optical and energetic characteristics of Transition-Metal Dichalcogenides (TMDs) in Graphene (Gr) VDW heterostructures, monolayer (Graphene, MoS_2 and WS_2), double layer and tetra layer systems. They discovered that whereas out-of-plane orbitals contribute weakly to hybridization in the TMD systems and increase the binding energy, the Gr–TMD interactions were controlled by VDW interactions.

The publication [14] presented the use of molecular beam epitaxy to incorporate magnetic manganese ions into $MoSe_2$ single-layer structures. They conduct growth tests on a variety of substrate with very disparate characteristics, included monocrystalline sapphire, exfoliating carbon (on tantalum foil), polymorphic Silicon dioxide (SiO_2) on silicon (Si) and exfoliated 2D hexagonal Boron Nitride flakes (placed on SiO2/Si).

The study [15] presented the successful epitaxial development via molecular beam epitaxy of a continuous, uniform, highly crystalline monolayer MoS_2 sheet over hexagonal boron nitride (h-BN). Research used electron microscopy with atomic force revealed that the two primary categories of nucleation grains in MoS_2 grown on h-BN were the 60° anti-aligned domain and the 0° aligned domain.

3. METHOD AND MATERIALS

We used 21 Independent Components (ICs) to minimize the dimensionality of the original dataset and speed up computing. We randomly selected a set of images to serve as the initial centroids, representing the starting points for cluster formation. The fuzzy C means a cluster of the dataset was divided into clusters by assigning each RHEED image to the closest centroid.

3.1 Data Processing and Analysis

On a substrate of (0001) 6H-Silicon carbide (SiC), an epitaxial graphene bilayer was formed and $ReSe_2$ thin films were generated using a custom-made MBE machine in ultrahigh vacuum (1.0×10^{-9} torr). The growth of the SiC substrate's double-layer carbon was aided by substrate outgassing for several hours at 649°C, as demonstrated by the RHEED image in Fig. 2f. Then, for six minutes, the substrates were annealed at 1330 °C. For the development of the $ReSe_2$ thin film, higher purity utilized was Re (99.9%) and Se (99.99%). we employed an in situ RHEED to monitor the film surface while co-evaporating Re and Se in a Knudsen cell and an electron-beam evaporator, respectively, to produce the $ReSe_2$ thin film, there was substrate. The ML-assisted growth analysis overview is shown in Fig. 1.

3.2 Characterization

A 532 nm excitation laser source was used to carry out the Raman spectroscopy experiments at room temperature with a fixed acquisition period and set power (60s). The scattered light from the materials was measured using a 50 cm focal length single-grating monochromatic and an instrument called a charge-coupled device detector, cooled by liquid nitrogen, picked up light. Following the statement of deposition, the surface morphology was examined using Atomic Force Microscopy (AFM) in an airtight environment. Using an NSC18/Pt tip, the non-contact mode of scanning was used on the samples. To investigate the films' stoichiometric, XPS measurements were performed. Cross-sectional specimens were created for the Scanning Transmission Electron Microscopy (STEM) analysis utilizing the concentrated ion beam method. All machines learning research was conducted using Python 3.10.11 and the model and code are available to the general public. First, the RHEED video was converted into an ICA-specific 2D array named X. This array was an O × P matrix, where O and P were the respective symbols for the number of pixels and frames. RHEED video was recorded at a frame-per-second frequency. This allowed every matrix row to represent a RHEED image at a specific point in time. Utilizing component weights, the dataset was transformed into a set that was linearly superposed for the ICA and the basis of orthogonal eigenvectors.

The P × P matrix was created using the basis matrix (boxes highlighted in red) and the individual ICs were represented by the column vectors. The components of the established matrix (the IC space's green-shaded boxes) were established based on the basis matrix and the manufacturing of X. The RHEED images were represented by the row vectors in the eigenvalues in descending order, or "Score," while the column vectors showed how each score changed over time.

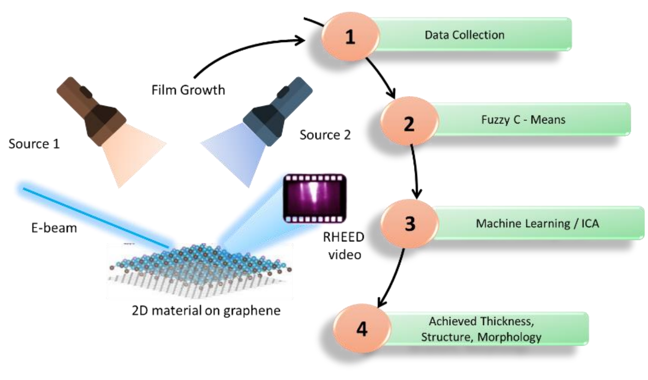

Fig. 1 – ML-assisted growth analysis overview

We proposed a reconstruction approach termed ICA, which entailed eliminating a few of the chosen ICs, like Original RHEED$-\sum_{j=1}^{m} IC_j$ and combining the IC space frames to eliminate contributions of the substrates. Next, we were able to determine the temporal dependences of the chosen diffract to reach their highest intensities, as the left bottom illustrates.

We employed 21 ICs to reduce the original dataset's dimension before performing the fuzzy C means cluster to speed up computing. Fuzzy C means clustering was applied to the RHEED image series and each image was grouped according to its closest mean, or "centroid." At first, the centroids were randomly selected images from the entire data collection. Finally, we designated each RHEED image's closest centroid.

4. RESULT

We generated a variety of thicknesses on $ReSe_2$ thin films on surfaces made of graphene. Fig. 2a shows the atomic composition of the deformed $ReSe_2$. Figs. 2b–d show the graphical representations of the substrate made of graphene and the $ReSe_2$ thin films, which have Unit Cells (UC) are 0.3 and 3, respectively. As seen in Figs. 2e–j, we contrasted the outcomes with data from AFM, using in situ RHEED measurements to monitor the development of $ReSe_2$. The double-layer carbon substrate was first made with a fat broad terrace on the surface (Fig. 2h) and an angular RHEED structure (Fig. 2e). After 4 minutes of film growth, more $ReSe_2$ lattice streaks became visible in the RHEED pattern; Fig. 2f's red signs indicate these. In Fig. 2f, red arrows indicate the RHEED pattern. Furthermore, the terrain developed small $ReSe_2$ islands (Fig. 2i). When the graphene was deposited for 1 hour, as seen in Fig. 2g, the complete disappearance of the RHEED pattern, left the $ReSe_2$ streaks. The $ReSe_2$ thin film's flat surface morphology was revealed by the vertically elongated $ReSe_2$streaks. The RHEED-shaped graphene streaks and $ReSe_2$ were compared to estimate the calculated lattice parameter in-plane of the $ReSe_2$ layer. The bulk value (6.60 Å(a1) and 6.71 Å(a2)) was consistent with the calculated lattice parameter in-plane of 6.58 Å. The resulting $ReSe_2$ thin film was predicted to have a thickness of around 3UC and a smooth surface that is rough at 0.23 nm (Fig. 2j). We used Raman spectroscopy to characterize the 3UC-thick $ReSe_2$, as seen in Fig. 2k. Because $ReSe_2$ breaks the inversion symmetry, $ReSe_2$ showed a range of vibration modes in the 100 – 300 cm $^{-1}$ range. The thickness affected the peak positions, which matched the characteristics of thick films and the bulk of $ReSe_2$. Furthermore, we assessed the layer thickness using Fig. 2 shows an examination use of High-Angle Annular Dark Field (HAADF) in STEM.

Three rows of white dots arranged horizontally bordered by grey dots are seen in this image, as indicated by the black arrows. The reduced signal in the top $ReSe_2$ layer suggests that the uppermost layer is not well covered. We looked at the $ReSe_2$ stoichio-metric using X-ray photo-emission spectroscopy (XPS). It was discovered that the atomic ratio of Se to Re was roughly 2.01, which was near to the stoichiometric ratio that was specified. Additionally, we measured the combined peak regions of Se 3d and Re 4f. These results validate the production of $ReSe_2$ thin layers with regulated thicknesses and indicate that ML techniques can be applied to the analysis of the accompanying RHEED data.

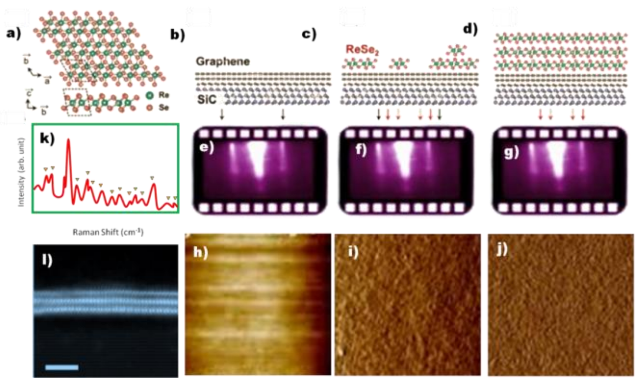

Fig. 2 (a-l) – Development and characterization of $ReSe_2$ thin films

Initially, we assessed the $ReSe_2$'s RHEED video. film using ICA. Fig. 3a and 3b display the first six Independent components (ICs) and the matching values for the scores. These concepts are analogous to eigenvalues and eigenvectors, in that order. The sum of the six parts is 99.85% of the dataset's variance in statistics, indicating that a limited number of components and scores can capture the majority of the dataset. Particularly, IC1 in the RHEED video has the highest volatility (92.97%). Fig. 3a shows two important aspects of the IC1. First, note how well the graphene pattern in Fig. 2e is matched by the positive (green) area. The (2, 0) and (− 2, 0) diffraction spots of $ReSe_2$ are located in the negative (red) region. As we go closer to the third dotted line in Fig. 3, we see a steady decline in IC1, or score 1, along with a sign transition from the positive to the negative.

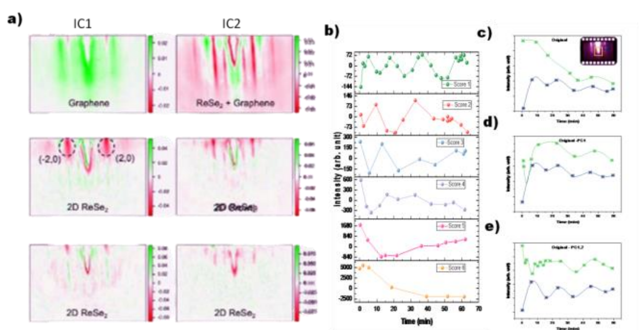

Fig. 3 (a-e) – ICA results

This implies that the primary observation in the first RHEED film is the graphene signal's dropping tendency. The oxide thin film signal pattern is very dissimilar from this one, where the lattice parameters in-plane is exactly matched. The second component, IC2, dominates $ReSe_2$ streaks and small diffraction sites on graphene and (Silicon carbide) SiC surfaces. Given the similar RHEED pattern of $ReSe_2$ in Fig. 2g, $ReSe_2$ thin film epitaxial 2D growth is represented by a negative value of IC2. Stained stripes in graphene and a few extra spaces in the middle are located in the positive (green) region of IC2. These areas match the SiC substrate underneath the graphene and the buffer layer above it. When score 2 (Fig. 3b) is reduced for the first time, the substrate pattern vanishes and the $ReSe_2$ pattern (first dashed line) starts to show. IC3-6 has the 3UC $ReSe_2$ layers' 2 and 2, 0 diffraction signals. The matching scores for the range of 3-6 show different patterns (Fig. 3b).

The involvement of graphene, which is positively connected with score 1, is shown by the (0, 0) peak intensity in Fig. 3c, which gradually decreases. As shown in Fig. 1b, we acquired the updated RHEED data (mICA) by deleting components connected to graphene (IC1 or IC2) based on the uncut RHEED footage to isolate the weak $ReSe_2$ signal from the original video. The streaks with blue lines (0, 0) and orange lines (2, 0) intensity maps acquired from the mICA video sets are shown in Fig. 3(d, e). The intensity plot in Fig. 3d is altered by the deletion of IC1 during the initial segment through the line with three dashes. This shift represents the graphene signal transition to $ReSe_2$, in line with the score 1 sign change (shown by an arrow in Fig. 3b. Additional subtraction of IC1 and IC2 in Fig. 3e produces steady oscillations for the blue and green curves. As indicated, the oscillating appearance of the (0, 0) and (2, 0) streaks is probably related to the development of the film layer by layer. The blue and green curves in Figure 3e exhibit continuous oscillating behaviors that yield precise information about the film thickness. As a result, the final film thickness of 3UC is in agreement with the STEM data shown in Fig. 2c. Thus, in Figs. 3b–e and 4a, we inserted the vertical dashed lines.

The clustering that is dependent on time and related centroids for each value is displayed in Figures 4a and b. More separated segments show up for the first growing period (i.e., less than 35 minutes) and they are increased from 2 to 6, suggesting that the significant pattern shift mostly happens at the original time frame. The vertical dashed lines and the borders between the groups are in excellent alignment (Fig. 4a). The legitimate number of clusters is determined by the cost function, which is the total of the clusters' differences and the original data, as illustrated in Fig. 4c. The acceptable located close to the curve's saturation point.

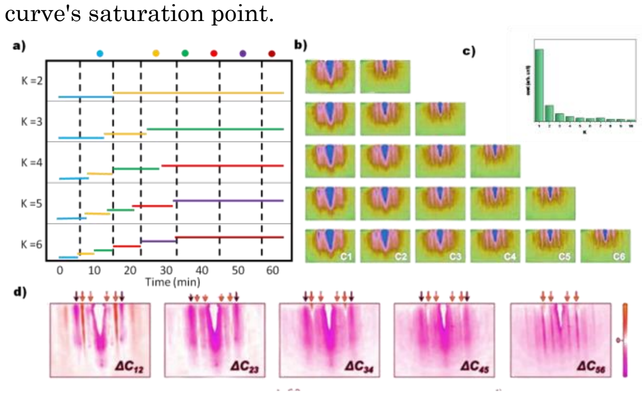

Fig. 4 (a-d) – C-means clustering analysis of the 3UC $ReSe_2$ RHEED video

When there is a saturation of the cost function. To further investigate the centroids' evolution, we deducted a prior centroid (C_i) from a subsequent one ($C_i + 1$), as shown in Fig. 4d and visualized the variation (ΔC_i ($i + 1$)) between the adjacent centroids. Red for the positive and pink for the negative portions in the RHEED patterns respectively, stand for the emerging and fading features. The growing $ReSe_2$ streak signal (shown by red arrows) in ΔC12 is a distinctive characteristic that matches the emerging $ReSe_2$ signal in the ICA. Up until ΔC45 (23 minutes), the graphene signal (black arrows) exhibits a trend that eventually disappears.

This barrier is represented by the third dashed line, which is where score 1 in the ICA turns negative and the graphene signal almost vanishes (Fig. 2b). ΔC56 primarily displays the intensity fluctuations in the $ReSe_2$ streaks after the graphene signal vanishes, suggesting a homoepitaxial development regime, because of outcomes of the ICA.

5. CONCLUSION

The use of Machine Learning (ML) facilitated Reflection (RHEED) High-Energy Electron Diffraction analysis in the study of dichalcogenides ($ReSe_2$) of transition metals thin-film formation on substrates made of graphene has demonstrated efficacy in comprehending the dynamics of film-growing. Different patterns in growth processes were found by applying Independent Component Analysis (ICA) and Fuzzy C-means clustering. By shedding light on the controlled deposition and crystallization of 2D materials, this study demonstrates the potential of machine learning approaches to revolutionize nanotechnology and device production. However, there are disadvantages such as the need for careful ML parameter adjustment and the reliance on a certain material system. To improve thin film production techniques, future research should investigate how well the ML approach generalizes to various metal dichalcogenides and substrates, address scalability that expand its applicability to a wide range of material systems. Real-time monitoring during deposition could be used to improve machine learning models for dynamic control of film growth.

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Аналіз зростання 2D тонких плівок перехідного металу на графенових підкладках на основі кластеризації методом молекулярно-променевої епітаксії

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Дихалькогеніди металів – це різновид хімічної речовини, яка складається з атома металу в парі з халькогеновими елементами, такими як селен і сірка. Ці матеріали мають відмінні електричні та оптичні характеристики, що робить їх захоплюючими для різноманітних застосувань, включаючи електроніку та оптоелектроніку. Дослідження росту тонких плівок дихалькогенідів металів передбачає аналіз їх контрольованого осадження та кристалізації. Розуміння процесів росту, взаємодії підкладки та контрольних параметрів, таких як температура та концентрація прекурсора, є критично важливими для виробництва високоякісних плівок із відповідними характеристиками, закладаючи шлях для розвитку нанотехнологій та виробництва пристроїв. У цьому дослідженні ми використовували аналітичний підхід дифракції високоенергетичних електронів із підтримкою машинного навчання (ML) для дослідження розвитку двовимірних (2D тонких шарів дихалькогенідів ($ReSe_2$) з перехідних металів на графенових підкладках за допомогою молекулярної Променева епітаксія (MBE). Незалежний аналіз компонентів (ICA) і підхід Fuzzy C-Means були реалізовані для визначення різних шаблонів і представлення розмірності вихідного набору даних, ми використовували 20 незалежних компонентів (IC) і кожне зображення RHEED. було розподілено до найближчого центроїда, що призвело до кластеризації набору даних за допомогою нечітких C-середніх.

Ключові слова: Аналіз росту, 2D дихалькогенід перехідного металу, Графенові підкладки, Молекулярно-променева епітаксія.