Marangoni-Effect-Driven Growth of Transition Metal Dichalcogenide Nanoribbon Arrays

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Abstract

The controlled synthesis of transition metal dichalcogenide (TMD) nanoribbon arrays represents a promising route for integrating two-dimensional (2D) semiconductors into large-scale circuits. However, in nanoribbon synthesis, the excessively large diameter of transition metal source droplets gives rise to dominant non-directional surface driving forces, which disrupt the directional migration of droplets and thereby hinder the growth of nanoribbon arrays. In this work, we exploit the Marangoni effect to provide the driving force for molten precursor droplets, while the atomic steps of sapphire substrates endow this force with directionality. The introduction of nickel reduces the droplet size by a factor of 100, which significantly enhances the effectiveness of this directional driving force. Based on this approach, we successfully guide the directional migration of molten transition metal precursor droplets, enabling the fabrication of highly aligned nanoribbon arrays. In situ visual chemical vapor deposition allows real-time observation of droplet-mediated growth, whereas density functional theory calculations and force analysis elucidate the underlying driving mechanisms. This work provides a mechanistic framework and a general synthetic strategy for the scalable preparation of well-ordered TMD nanoribbon arrays suitable for next-generation 2D semiconductor integration.

Full Text

Preamble

Marangoni Effect Driven Growth of Transition Metal Dichalcogenides Nanoribbon Arrays

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Keywords: transition metal dichalcogenides, nanoribbon arrays, chemical vapor deposition, field-effect transistors

Abstract: The controlled synthesis of transition metal dichalcogenide (TMD) nanoribbon arrays represents a promising route toward integrating two-dimensional (2D) semiconductors into large-scale circuits. However, in nanoribbon synthesis, excessively large diameters of transition metal source droplets lead to dominant non-directional surface driving forces, which disrupt directional droplet migration and consequently hinder the growth of nanoribbon arrays. In this work, we leverage the Marangoni effect to provide a driving force for the molten precursor droplets, while the atomic steps of the sapphire substrate impart directionality to this force. The introduction of nickel reduced the droplet size by a factor of 100, which significantly enhanced the efficacy of this directional driving force. Based on this approach, we successfully guided the directional migration of molten transition metal precursor droplets, thereby enabling the fabrication of highly aligned nanoribbon arrays.

In situ visual chemical vapor deposition enables direct observation of droplet-mediated growth, while density functional theory calculations and force analyses elucidate the underlying driving mechanisms. This work provides both a mechanistic framework and a versatile synthesis strategy for the scalable fabrication of well-aligned TMD nanoribbon arrays suitable for next-generation 2D semiconductor integration.

Introduction

Two-dimensional (2D) transition metal dichalcogenides (TMDs) possess atomic-scale thickness, dangling-bond-free surfaces, and excellent electronic and optical properties, rendering them ideal for high-performance nanoelectronic and optoelectronic applications.[?] Tailoring their dimensionality and morphology further expands their functional scope.[?, ?] In particular, one-dimensional TMD nanoribbons exhibit distinctive quantum confinement and edge-induced phenomena, leading to unique physical properties[?] and high carrier mobilities[?] arising from their anisotropic electronic structures. These characteristics make TMD nanoribbons promising candidates as channel materials in field-effect transistors (FETs).[?, ?] Their well-defined transport pathways and reduced edge scattering enable FETs with superior carrier mobility and switching performance.[?] Aligned nanoribbon arrays with large aspect ratios represent an ultimate form of lateral miniaturization, making them essential building blocks for dense FET integration.[?] Synthesis control is critical for exploring and exploiting the intrinsic properties of TMD nanoribbons. Two general approaches have been developed: top-down[?, ?, ?] and bottom-up.[?, ?] The top-down route, which relies on lithographic patterning or electron-beam etching, offers precise pattern definition but suffers from complex processing steps and limited edge quality.[?] In contrast, bottom-up strategies enable direct crystal growth, offering superior uniformity and structural precision.[?] For example, Li et al. demonstrated vapor-liquid-solid growth, where molten precursor droplets migrate across the substrate to form nanoribbons.[?] Substrate engineering has also been exploited to promote guided epitaxy, enabling the formation of nanoribbons on β-Ga_2O_3,[?] Au,[?] and sapphire substrates.[?] Xue et al. further showed that step edges on annealed sapphire can direct the motion of molten droplets, yielding highly aligned WS_2 nanoribbon arrays.[?] However, a universal, controllable method for fabricating ordered nanoribbons remains elusive.

In particular, molybdenum-based TMDs pose a significant challenge due to the large size of their molten droplets, which diminishes the directional driving force required for guided motion, thus hindering ordered array formation.[?] Here, we report a general bottom-up strategy for synthesizing aligned nanoribbon arrays of various TMDs (MoS_2, $MoSe_2$, WS_2, WSe_2) via a Marangoni-effect-driven process. The atomic steps created on the sapphire substrate via high-temperature annealing impart a directional driving force to the transition metal source droplets, while the introduction of nickel ions into the precursor reduces droplet size, thereby enhancing the efficacy of this force and promoting directional migration. Using an in situ high-temperature visual chemical vapor deposition (CVD) platform, we directly captured, for the first time, the collective motion of Marangoni-driven droplets and their transformation into aligned nanoribbons. Complementary density functional theory (DFT) calculations and force analyses elucidate the mechanistic principles governing droplet migration and nanoribbon formation.

Our findings provide a universal synthesis pathway for large-scale, well-aligned TMD nanoribbon arrays, offering a scalable foundation for sub-nanometer integrated 2D electronics.

Results and Discussion

Growth of The Nanoribbon Arrays

To achieve the controlled synthesis of TMD nanoribbon arrays, we implemented a dual strategy in which engineered atomic steps on sapphire substrates[?] provided a directional driving force, while nickel incorporation reduced precursor droplet diameter to enhance step guidance (Figure 1a [Figure 1: see original paper]).

Growth was carried out on C-plane sapphire substrate with a 1° miscut toward the M-axis (C/M-1°).[?] The precursor solution, composed of Na_2MoO_4 and

$NiCl_2$ dissolved in deionized water, was deposited onto the substrate using a precision dispensing system equipped with a micrometer-scale nozzle (Figure S1). This technique allowed accurate spatial control over precursor placement and enabled multiple growth sites to be patterned simultaneously.[**?**] Unlike previous water-vapor-assisted volatilization-deposition methods employing nickel nanoparticles,[**?**] we directly introduced Ni^{2+} ions into the precursor solution, allowing fine-tuned adjustment of nickel concentration and, consequently, droplet size and mobility.

After precursor deposition, the substrate was loaded into a dual-zone tubular furnace for CVD. Detailed growth parameters are provided in the Experiment Methods section. The resulting TMD nanoribbons exhibited highly uniform alignment (Figure 1b), with lengths exceeding 50 μm (up to 262 μm; Figure S2) and widths below 200 nm. Raman and photoluminescence (PL) spectra confirmed both the high crystallinity and monolayer nature of the $MoSe_2$ nanoribbons (Figure S3). Raman mapping further verified the structural continuity and exceptional parallel alignment of the nanoribbon arrays (Figure 1c). Atomic force microscopy (AFM) revealed that the nanoribbons grew strictly along the substrate step edges (Figure 1d and Figure S4). Aberration-corrected scanning transmission electron microscopy (STEM) images showed a pristine hexagonal honeycomb lattice in the $MoSe_2$ nanoribbons (Figure 1e), further evidencing their excellent crystallinity. Owing to this structural perfection, the fabricated FET exhibits superior electrical performance, including a carrier mobility of ~4 $cm^2\ V^{-1}\ s^{-1}$ and an on/off ratio of ~10^6 (Figure 1f).

Growth Mechanism of The Nanoribbon Arrays

For nanoribbon array synthesis, the key process is the directional migration of molten sodium molybdate droplets across the substrate surface.[**?**] Droplets spontaneously migrate[**?**] from regions of lower surface free energy to those with higher surface free energy,[**?**, **?**] and this surface tension gradient-induced migration is known as the Marangoni effect.[**?**] DFT calculations determined the specific surface free energies of $MoSe_2$ and the c-plane (0001) of sapphire (Figure 2e [Figure 2: see original paper]). The sapphire (0001) plane exhibits a much higher specific surface energy (~4.96 J/m^2) than $MoSe_2$ (~2.87 J/m^2). Based on this, molten droplets spontaneously move toward regions of sapphire substrate surface, thereby driving the anisotropic formation of $MoSe_2$ nanoribbons (Figure 2a and 2b). The growth process occurs within 10 seconds, and the nanoribbons are mostly curved, consistent with previous reports.[**?**, **?**, **?**]

An analysis of the mechanical forces exerted on the droplet was conducted.[**?**, **?**] The wetting behavior of droplets at the three-phase contact line is described by Young’ s equation:[**?**] where γ{*sv*}*,* γ{sl}, and γ_{lv} represent interfacial tensions at the solid-vapor, solid-liquid, and liquid-vapor interfaces, respectively, and θ is the droplet contact angle. As the $MoSe_2$ layer forms, a surface tension gradient develops at the $MoSe_2$/sapphire interface, providing the driving force for droplet motion. However, because of the isotropy of the step-free substrate, the resulting driving force is non-directional. This inherent variability ultimately results in the random orientation observed in the growing nanoribbons.

To achieve parallel alignment, we introduced periodic atomic steps on the sapphire substrate through high-temperature annealing of the C/M~1° sapphire (Figure S7).[?] Each step comprises a riser face (M-plane of sapphire) and a tread face (C-plane of sapphire). DFT calculations reveal that the (10 10) plane exhibits a higher surface free energy (~6.56 J/m^2) than the (0001) plane, establishing a surface energy difference that exerts a directional driving force on the droplet. The droplet interacts with the densely spaced step risers of the sapphire (10 10) plane, which have a well-defined driving force with a fixed orientation. As a result, the spontaneous migration of the droplet is constrained by this additional force, thus moving parallel to the steps (Figure S6b). AFM (Figure 2c) and visual CVD imaging (Figure 2d) confirm that nanoribbons preferentially align and grow along these step edges. Statistical analysis (Figure 2f) shows that over 95% of the nanoribbons grow in a single direction on the stepped surface, compared with only 2% on step-free surface.

Besides the steps, nickel incorporation plays a crucial complementary role in achieving well-aligned nanoribbon arrays (Figure S8). Introduction of nickel markedly reduces the diameter of molten precursor droplets,[?] thereby enhancing their ability to respond to the step-induced directional force. Auger electron spectroscopy mapping (Figure S9) and TEM-EDS analyses (Figure S10) confirmed the presence of nickel in terminal particles at the nanoribbon ends. As shown in Figure 3a [Figure 3: see original paper], increasing $NiCl_2$ concentration systematically reduces terminal particle size. The results indicate that without nickel, the terminal particles typically exhibit diameters exceeding 900 nm. With the addition of 0.025 mg/mL nickel chloride, the diameters are reduced to greater than 400 nm, while at 0.05 mg/mL, they are reduced to less than 200 nm. Statistical analysis revealed that the absence of termination particles was observed in over 30% of the nanoribbons, attributed to the depletion of the metal source. Furthermore, the data from Figure 3h reveal a correlation between the diameter of the molten droplets and that of the terminal particles left after growth. This demonstrates that an appropriate concentration of nickel chloride effectively reduces the diameter of the molten droplets.

A decrease in the droplet diameter leads to a change in the driving force. We define the molten droplet diameter without nickel addition as D_0, and that with nickel addition as D_{Ni}. For both conditions, the driving forces acting on the droplet—originating from the c-plane tread face and the m-plane riser face of the step—can be determined (Figure S11). Given that the droplet diameter is much larger than the step height, the driving force arising from the surface free energy difference between the m-plane sidewall of the step and the $MoSe_2$ surface remains essentially constant under both conditions. In contrast, the isotropic driving force lacks a defined direction, whereas the step-induced driving force acts parallel to the step riser. When the droplet diameter falls below approximately 200 nm, the influence of the directional driving force increasingly dominates over the isotropic force. This dominance induces the collective, aligned migration of droplets, ultimately leading to the formation of well-ordered nanoribbon arrays.

Through nickel modulation, the nanoribbon width can be precisely tuned to 200 nm, 100 nm or 50 nm (Figure 3b-g). Statistical analysis (Figure 3k) shows that only 10% of the nanoribbons grew along the step direction in the absence of $NiCl_2$, thereby confirming its critical role in the process. However, further increasing the $NiCl_2$ concentration gradually shortens the nanoribbons and eventually suppresses their growth entirely (Figure S12). In summary, the combined effects of substrate step construction and droplet size regulation govern the direction and magnitude of the Marangoni-driven force, enabling the controlled synthesis of TMD nanoribbon arrays.

Syntheses of Other TMDs Ribbon Arrays

Having established the synthesis of $MoSe_2$ nanoribbons, we next sought to demonstrate the generality of our dual growth strategy by extending it to other TMDs, including MoS_2 (Figure 4a [Figure 4: see original paper]), WS_2 (Figure 4b), and WSe_2 (Figure 4c). While the overall growth process remained analogous, specific adjustments were made for tungsten-based systems, employing Na_2WO_4 as the precursor and carefully optimizing process parameters for each material. To unambiguously confirm the chemical composition and crystalline quality of the resulting nanoribbons, we conducted Raman spectroscopy.

The MoS_2 nanoribbons exhibited the characteristic E^2g and A_1g vibrational modes at 382 cm^{-1} and 410 cm^{-1}, respectively (Figure 4d). Likewise, WS_2 nanoribbons displayed corresponding peaks at 355 cm^{-1} and 420 cm^{-1} (Figure 4e), while the pronounced A_1g mode at 250 cm^{-1} verified the successful synthesis of WSe_2 (Figure 4f). PL spectroscopy further confirmed the monolayer nature and direct bandgap emission of each material. Distinct PL peaks were observed at 676 nm for MoS_2, 631 nm for WS_2, and 770 nm for WSe_2 (Figure S13), consistent with the expected quantum confinement behavior of monolayer semiconductors. Definitive structural evidence was obtained through aberration-corrected STEM. The images shown in Figures 4g-i clearly reveal pristine hexagonal honeycomb lattices, while the corresponding selected-area electron diffraction (SAED) patterns can be precisely indexed to their respective crystal structures. Collectively, these results confirm not only the successful synthesis of the target TMD nanoribbons but also their exceptional crystalline quality and structural uniformity across multiple compositions.

Conclusions

In summary, we have developed a general and scalable strategy for the synthesis of TMD nanoribbon arrays. This method integrates two synergistic elements that combine sapphire step-guiding with nickel doping, utilizing the Marangoni effect to drive the directional migration of precursor droplets. The atomic steps on sapphire substrates generate a parallel driving force for the droplets. The introduction of nickel reduces the diameter of the precursor droplets, making them more susceptible to this directional guidance. Consequently, by controlling the directional movement of the droplets, we achieved the synthesis of aligned TMD nanoribbon arrays. The alignment and parallel orientation of the arrays were confirmed by Raman mapping and AFM. Furthermore, the high crystallinity and quality of the nanoribbons were verified by Raman spectroscopy, PL, and electron microscopy characterizations. These findings not only provide valuable insights into the underlying growth mechanisms but also highlight the versatility of this approach for the large-scale fabrication of TMD nanoribbon arrays for applications in next-generation 2D electronic devices.

Associated Content

Supporting Information: Experimental details including sapphire substrate preparation, precursor printing, Raman and PL spectroscopy, process parameter optimization, in-situ visualization, elemental analysis, growth characterization, and theoretical computation methodologies. (docx)

Supporting Video 1: In Situ Observation Data of Growth on Step-free Substrate Surface (avi)
Supporting Video 2: In Situ Observation Data of Growth on Stepped Substrate Surface (avi)
Supporting Video 3: In Situ Observation Data of Droplet-mediated Growth of A Single Nanoribbon (avi)
Supporting Video 4: In Situ Observation Data of The Precursor Heating Process (avi)
Supporting Video 5: In Situ Observation Data of Growth without $NiCl_2$ Addition (avi)

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Notes: The authors declare no competing financial interest.

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Abbreviations

TMD, transition metal dichalcogenide; 2D, two-dimensional; FET, field-effect transistor; CVD, chemical vapor deposition; DFT, density functional theory; STEM, scanning transmission electron microscopy; SAED, selected-area electron diffraction; AFM, atomic force microscopy

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Note: Figure translations are in progress. See original paper for figures.