Robust growth of two-dimensional metal dichalcogenides and their alloys by active chalcogen monomer supply

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The precise precursor supply is a precondition for controllable growth of two-dimensional (2D) transition metal dichalcogenides (TMDs). Although great efforts have been devoted to modulating the transition metal supply, few effective methods of chalcogen feeding control were developed. Here we report a strategy of using active chalcogen monomer supply to grow high-quality TMDs in a robust and controllable manner, e.g., MoS$_2$ monolayers perform representative photoluminescent circular helicity of ~92% and electronic mobility of ~42 cm$^2$/V s. Meanwhile, a uniform quaternary TMD alloy with three different anions, i.e., MoS$_2$(1-x-y)Se$_2$xTe$_2$y, was accomplished. Our mechanism study revealed that the active chalcogen monomers can bind and diffuse freely on a TMD surface, which enables the effective nucleation, reaction, vacancy healing and alloy formation during the growth. Our work offers a degree of freedom for the controllable synthesis of 2D compounds and their alloys, benefiting the development of high-end devices with desired 2D materials.
Two-dimensional (2D) transition metal dichalcogenides (TMDs), with their atomic thicknesses, high carrier mobility, fast charge transfer, and intrinsic spin-valley couplings, have been demonstrated one of the most appealing candidates for next-generation electronic and optoelectronic devices\textsuperscript{1–3}. The wafer-scale synthesis of TMDs with well-controlled crystallinity, quality, and composition is essential to fully realize their promising applications\textsuperscript{4–13}. However, it is well known that the controllable growth of multi-element bulk materials is generally much more challenging than that of single-element ones. For example, the synthesized first-generation semiconductor, silicon, can have the extremely low impurity of ~10\textsuperscript{-11} and is nearly threading dislocation free, but the synthesized third-generation semiconductor, GaN, generally has a much higher impurity, ~10\textsuperscript{-4}, and a threading dislocation density of ~10\textsuperscript{8}–10\textsuperscript{10} cm\textsuperscript{-2} (ref. 14). Analogously for the growth of 2D materials, the as-grown graphene already has excellent properties, which is comparable to the samples exfoliated from natural crystals, and the measured carrier mobilities are close to the theoretical limit\textsuperscript{15}, while the as-grown 2D compounds of TMDs, typically have lower quality than the natural crystals or the theoretical expectations\textsuperscript{16}. The main difficulty in controllable compounds’ growth lies in the complicated feeding of several elements simultaneously during the growth process. Therefore, in the semiconductor industry, advanced and expensive techniques such as molecular beam epitaxy (MBE) and metal-organic chemical vapour deposition (MOCVD) have been developed to realize the precise control of multi-element supplies for the compound film growth.

Similarly, the synthesis of high-quality TMD materials requires the precise feeding control of both the transition metal and chalcogen precursors. In the past decade, intensive efforts have been devoted to optimizing the feeding of metal precursors by thermal evaporation or molten-salt-assisted evaporation of metal oxide\textsuperscript{17}, decomposition of metal-organic precursors\textsuperscript{18}, direct deposition of metal layers, and others\textsuperscript{19–21}. Although some methods for controllable chalcogen feeding, such as using either elemental chalcogen or chalcogen compounds (i.e., heating sulfur powder, using H\textsubscript{2}S gas and ammonium sulfide\textsuperscript{22–24}), have also been developed, it turns out that the chalcogen feeding control is much less effective than metal feeding control, as indicated by the most challenging problem in TMD quality control that the most synthesized TMDs are rich with chalcogen vacancies\textsuperscript{25}. Therefore, developing more effective chalcogen supply methods to enable the growth of TMDs with high quality and rich composition is of critical importance.

In this work, we propose to use a chalcogen monomer feeding method in the controllable TMD growth because of the following potential advantages. (i) The chalcogen monomers or atoms are generally very active than the corresponding dimers or bulks and thus they can quickly react with metal precursors to form TMDs, (ii) the active chalcogen monomers can bond and quickly diffuse on a TMD surface to scavenge the vacancy defects effectively, which will greatly improve the quality of the TMDs and (iii) an active chalcogen monomer can react with a TMD and easily substitute a chalcogen atom and, thus, allow the synthesis of uniform TMD chalcogen alloys. However, as the monomer state of chalcogen only exists at very high temperature (>2500 K) under normal circumstance\textsuperscript{26}, the most used methods cannot produce enough chalcogen monomers at the typical TMD growth temperature, which is generally ~1300 K. Herein, we developed an effective route to provide chalcogen monomer by heating a metal chalcogenide. The success of this approach lies in that the chalcogen atoms on the surface of metal chalcogenides can be easily released in the chalcogen atom (monomer) form under a relatively lower temperature\textsuperscript{27,28}. The slowly released chalcogen monomers have a very low probability to react with each other to form dimers, thus, enable the successful synthesis of TMDs (MX\textsubscript{2}, M = Mo, W; X = S, Se, Te) and their alloys with very high quality.

**Results**

**MoS\textsubscript{2} growth by sulfur monomer supply.** In our design, the metal chalcogenide plate of ZnS, Na\textsubscript{2}MoO\textsubscript{4} coated silica fibre fabric and the target substrate were vertically stacked as a sandwich structure by using mica spacers (Fig. 1a, left panel). The distance of d\textsubscript{1} (d\textsubscript{2}) between the S (Mo) precursor and the growth substrate can be modulated by varying the thickness of mica (from tens to hundreds of microns), which is essential to tune the fluxes of S and Mo independently. At an elevated growth temperature (~750–950 °C), S monomers were released from the ZnS surface and Na\textsubscript{2}MoO\textsubscript{4} started to vapourize (Fig. 1a, middle panel). Then both S and Mo sources passed through the porous fibre fabric (Supplementary Fig. 1) and reached to the substrate surface to form monolayer MoS\textsubscript{2} (Fig. 1a, right panel). It is important to note that the S monomers can’t be obtained by the sublimation of sulfur powders, where S\textsubscript{2} dimers are always the dominating species at thermal equilibrium\textsuperscript{29}. Indeed, the release of S monomers from the metal sulfide surface was observed long time ago\textsuperscript{30}, and was unambiguously revealed by the in-situ mass spectroscopy as shown in Fig. 1b.

In our experiment, we found that the flux of S monomers is significantly larger than that of the Na\textsubscript{2}MoO\textsubscript{4} or the S is overfed (Fig. 1c and Supplementary Note 1), as we observed that the variation of d\textsubscript{1} (the distance between the Na\textsubscript{2}MoO\textsubscript{4} and the substrate) has limited effect on the MoS\textsubscript{2} growth, while the variation of d\textsubscript{2} (the distance between the fibre fabric and the substrate) affects the nucleation density of MoS\textsubscript{2} domains greatly (Supplementary Fig. 2). This phenomenon can be qualitatively understood as that, by decreasing the d\textsubscript{2}, the landing probability of evaporated Mo source onto the substrate becomes larger (Fig. 1d and Supplementary Note 2), which can further boost the nucleation of MoS\textsubscript{2} domains. As shown in Fig. 1e, f, sparse triangular domains and 2-inch continuous film of MoS\textsubscript{2} can be respectively obtained after the same growth duration of 50 min when d\textsubscript{2} was 100 μm and 20 μm. In the limit case, the d\textsubscript{2} can also be zero by directly precoating a thin layer of Na\textsubscript{2}MoO\textsubscript{4} precursor into the substrate (Supplementary Fig. 3).

**Quality characterizations of MoS\textsubscript{2} domains.** A series of characterizations unequivocally reveal the high quality of MoS\textsubscript{2} fed by S monomer supply. Atomic-resolved atomic force microscopic (AFM) images at different positions of the as-grown MoS\textsubscript{2} clearly resolved the S atoms without obvious S vacancies (Supplementary Fig. 4). High-angle annular dark-field scanning transmission electron microscopic (HAADF-STEM) images exhibited the perfect hexagonal honeycomb lattice with both Mo and S atoms (Fig. 2a and Supplementary Fig. 5). And the density of S vacancy defects was extracted as ~2 × 10\textsuperscript{12} cm\textsuperscript{-2}, which is among the lowest value in previous STEM measurements (Supplementary Table 1).

The low-temperature photoluminescence (PL) spectra of the S-monomer-feeding-grown MoS\textsubscript{2} (Fig. 2b, orange curve) has a characteristic neutral exciton (X\textsuperscript{0}) emission peak accompanied with a trion (X\textsuperscript{T}) peak (believed to be caused by the n-type doping from substrate\textsuperscript{31}), but the X\textsuperscript{0} peak (believed to be caused by S vacancy\textsuperscript{32} and was obvious in S-powder-feeding-grown samples, Fig. 2b, dark yellow curve) is nearly invisible, which clearly proves the high quality of the S-monomer-feeding-grown MoS\textsubscript{2} samples. Meanwhile, the uniform distribution of the PL peak intensity and the narrow full width at half maximum both
demonstrated the high uniformity and crystallinity of the sample grown by S monomer supply (Supplementary Fig. 6).

In addition, the high quality of as-grown MoS2 can be further confirmed by the measured optical circular helicity33, which was detected to be as high as 92% (Fig. 2c) and comparable with the best-exfoliated flakes from high-quality natural crystals. The circular helicity is directly related to the scattering between K and K’ valleys in the Brillouin zone of MoS2 whilst the defects will greatly enhance the inter-valley scattering and decrease the circular helicity value. Thus, the near-unity circular helicity strongly proves the high quality of the as-grown MoS2 samples.

We further verified the quality of the single-crystal MoS2 domain by evaluating its field-effect mobility in a bottom-gate transistor configuration. The output and transfer characteristics of a typical field-effect transistor (FET) devices with channel length/width (L/W) of 7/22 μm are exhibited in Fig. 2d and Supplementary Fig. 7. The device exhibits a typical n-type transfer characteristic with an on/off ratio of ~108 at room temperature and electron field-effect mobilities of ~42 cm²V⁻¹s⁻¹, which are comparable to the performance of monolayer MoS2 prepared by mechanical exfoliation34. Statistics of the transport measurement are also given in Supplementary Fig. 8, demonstrating the good uniformity of the MoS2 electronic devices. These results suggest that S-monomer-feeding-grown MoS2 samples have appreciably high electronic quality.

Universal TMD growth by chalcogen monomer supply. Our strategy on MoS2 growth by monomer feeding has also been proved to be applicable for the growth of various high-quality TMD materials. Six typical monolayer TMDs (Fig. 2e) have been successfully synthesized by simply replacing the transition metal sources (e.g., Na₂MoO₄ and Na₂WO₄) and chalcogenide plates (e.g., ZnS, ZnSe, and ZnTe) (see Methods and Supplementary Fig. 9). The Raman and PL spectra of these obtained TMD samples demonstrated the successful synthesis of the 2H phase MoS2, WS2, MoSe2, WSe2, MoTe2, and the 1T phase WTe2 and MoTe2 (Supplementary Fig. 10). It is worth noting that, due to their higher formation energies in relative to the corresponding sulfide and selenide bulks (Fig. 2f and Supplementary Note 3), the growth of transition metal tellurides, e.g., MoTe2 and WTe2, by using chalcogen bulks as feedstocks is usually very challenging35. Thanks to the introduction of active Te monomers, the synthesis of WTe2 and MoTe2 is very easy and efficient because of the greatly reduced formation energies (Fig. 2f).

Controllable synthesis of quaternary TMD alloy. The chalcogen monomer feeding method has a unique advantage in the growth of TMD alloy. Since the evaporation temperatures, saturated vapour pressures and reaction energies of S, Se, and Te are significantly different, it is nearly impossible to form high-quality TMD alloys with more than two anion elements by traditional CVD approaches36. Till now, there is no report on the successful growth of MoS₂ₓ(1-x-y)Se₂ₓTe₂₀ alloy. In our experiment, we applied a compressed plate mixed with different metal chalcogenide powders, i.e., ZnS, ZnSe, and ZnTe, to supply three kinds of chalcogen monomers (S, Se, and Te) simultaneously (Fig. 3a). The as-grown alloy of MoS₂ₓ(1-x-y)Se₂ₓTe₂₀ has a triangular domain similar to 2H phase TMDs (Fig. 3b). The X-ray photoelectron spectroscopy (XPS) unambiguously revealed the coexistence of S, Se, and Te atoms in the synthesized TMD alloy (Supplementary Fig. 11). The energy-dispersive X-ray spectroscopy (EDS) as well as the STEM measurements further demonstrated the homogenous element distribution throughout the TMD alloy in both macro- and micro-scales, with no observable phase separation (Fig. 3e and Supplementary Fig. 12). Enlarged STEM image (Fig. 3f) further demonstrated the high...
crystallinity of the TMD alloy. The distinct intensity distribution revealed the occupancies of Mo, S, Se, and Te according to the Z-contrast nature of HAADF image (Fig. 3g). Quantitative analysis of the Te and Se distribution is presented in a 32 × 32 nm² STEM image, and the statistical results match well with the binomial distribution model (Fig. 3h, i, and Supplementary Fig. 13), suggesting a random distribution of S, Se and Te atoms in the TMD alloy.

Furthermore, one can easily tune the composition of the TMD alloys by controlling the growth temperature to vary the fluxes of S, Se, and Te from the ZnS, ZnSe, and ZnTe composite. As the growth temperature increased, the PL peak shifted to longer wavelength (Fig. 3c), revealing the increase of the concentrations of Se and Te in the alloy (MoSe₂ and MoTe₂ have smaller bandgaps than MoS₂). Meanwhile, in the Raman spectra, the MoS₂-featured peaks gradually attenuated, the MoTe₂-featured peaks gradually enhanced, while the MoSe₂-featured peaks enhanced first and then attenuated (Fig. 3d), which indicates that heavier chalcogen atoms are being doped into the alloy at a higher temperature.

**Mechanism for the chalcogen monomer-modulated TMD growth.** Finally, we try to understand the unique role of chalcogen monomer supply in the synthesis of high-quality TMDs and their complex alloys theoretically. We firstly explore the reactions of Na₂MoO₄ with sulfur monomers and dimers, respectively, by first-principles molecular dynamic (MD) simulations (Supplementary Fig. 14). The simulation results clearly demonstrated that sulfur monomers are more reactive to substitute the oxygen atoms in a MoO₄²⁻ group of Na₂MoO₄ while most dimers are desorbed from the Na₂MoO₄ surface due to their less activity (Supplementary Note 5). By adding more S monomers and MoS₂ clusters to the Na₂MoO₄, the nucleation of MoS₂ clusters can also be clearly seen (Supplementary Fig. 15).

In compare with the bond-saturated S₂/Se₂/Te₂ dimers, S/Se/Te monomers possess much higher adsorption energies on a TMD surface (for dimers Eₐ < 0.8 eV, for monomers Eₐ > 1.5 eV) (Fig. 4b and Supplementary Fig. 16a). Therefore, one can expect a large number of S/Se/Te monomers to diffuse on the TMD surface during the whole growth process and the growth of TMD is in a chalcogen monomer rich environment. Once a chalcogen monomer diffuses to the vicinal area of a vacancy, the vacancy can be quickly healed by a highly exothermic reaction (Fig. 4a, c and Supplementary Fig. 16b). Therefore, the chalcogen vacancy density is greatly reduced as it has been shown in the experimental results. To address the capacity of forming TMD chalcogen alloys, we calculated the reaction energy of substituting a chalcogen atom in a TMD by using chalcogen monomer, dimer, and bulk as references (Supplementary Fig. 17 and Supplementary Note 6). It is clearly shown that doping of Te dimer or bulk into MoS₂/MoSe₂, S bulk into MoSe₂, Se bulk into MoS₂ are all difficult because of the near-zero or positive reaction energies. While, if chalcogen monomer is used as the source of dopant, all doping reactions become exothermic with noticeable negative reaction energies, which implies the greatly improved capacity of forming chalcogen TMD alloys.

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**Fig. 2** Quality characterizations of the as-grown MoS₂ domain and universal growth of diverse TMDs by chalcogen monomer supply. a Atomic-resolved HAADF-STEM image of the prepared MoS₂, revealing the high crystallinity of MoS₂ without detectable S vacancies. b Low-temperature (10 K) PL spectra of MoS₂ samples fed by S monomer (orange curve) and S powder (dark yellow curve), respectively. Three typical features, X₀, Xᵀ, and XΔ peaks assign to neutral exciton, trion, and defect state emission peaks, respectively. The absence of XΔ peak confirmed the high quality of the MoS₂ grown by S monomer supply. c The circular dichroism PL spectrum measured at 10 K. The near-unity polarization of MoS₂ on sapphire indicates the high optical quality. The horizontal dashed line is added for clarity. d Transfer characteristic of the MoS₂ FET with channel length and width of 7 μm and 22 μm, respectively, at a bias voltage Vgs of 1 V. Inset: optical image of the device. e Optical images of the representative TMDs, including 2H phase MoS₂, MoSe₂, MoTe₂, WS₂, WSe₂ and 1T’ phase WTe₂. f The calculated formation energy (Eₐ) of the six representative TMDs. When chalcogen bulk is supplied as precursors, the formation of transition metal tellurides in relative to their corresponding sulfide and selenide are less favourable due to their high formation energies (−0.73 and −0.68 eV/unit for MoTe₂ and WTe₂, respectively). While it becomes highly favourable when Te monomers are applied.

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**Table 1** Calculated formation energy (Eₐ) of diverse TMDs calculated by the DFT simulation. a Reaction energy (eV) of the six representative TMDs. When chalcogen bulk is supplied as precursors, the formation of transition metal tellurides in relative to their corresponding sulfide and selenide are less favourable due to their high formation energies (−0.73 and −0.68 eV/unit for MoTe₂ and WTe₂, respectively). While it becomes highly favourable when Te monomers are applied.
Discussion
This study clearly demonstrated that the high reactivity of chalcogen monomers can significantly facilitate the TMD nucleation, chalcogen defect healing in the growth to greatly improve the samples’ quality and allow the formation of various chalcogen TMDs and their alloys. The monomer supply should provide a degree of freedom in modulating compound materials and high-TMDs and their alloys. The monomer supply should provide a degree of freedom in modulating compound materials and high-TMDs and their alloys.

Methods
Growth of TMDs and their alloys. The substrate (sapphire or SiO2/Si) and silica fibre fabric were first pretreated with O2 plasma. Then the silica fibre fabric was immersed in Na2MoO4 or Na2WO4 aqueous solution with optimized concentrations (Na2MoO4 of 12, 18, and 6 mg/mL for MoS2, MoSe2, and MoTe2 growth, Na2WO4 of 20, 30, and 12 mg/mL for WS2, WSe2, and WTe2 growth, respectively). After dried in Ar atmosphere, silica fibre fabric and the chalcogenide crystal plate (ZnS, ZnSe, or ZnTe) were placed above the substrate in sequence by using two chambers of furnace was crushed with Ar (100 sccm) and heated to the optimized growth temperature (~780 °C, ~930 °C, ~800 °C, ~820 °C, ~750 °C, ~800 °C, and ~780 °C for MoS2, WS2, MoSe2, WSe2, 2H-MoTe2, 1T-MoTe2, and 1T-WTe2, respectively). During the growth process, the system pressure was kept at ~120 Pa and the growth duration was set as 10-60 min. After growth, the system was naturally cooled down to room temperature. Similar growth conditions were optimized growth temperature (~780 °C, ~930 °C, ~800 °C, ~820 °C, ~750 °C, ~800 °C, and ~780 °C for MoS2, WS2, MoSe2, WSe2, 2H-MoTe2, 1T-MoTe2, and 1T-WTe2, respectively). During the growth process, the system pressure was kept at ~120 Pa and the growth duration was set as 10-60 min. After growth, the system was naturally cooled down to room temperature. Similar growth conditions were applied to the TMD alloy growth, wherein the major difference lies in the use of a chalcogenide mixture plate.

Sample characterization. Optical images were taken with an Olympus BX51M microscope. Raman and PL spectra were measured by a customer-designed optical system with the laser excitation wavelength of 532 nm and the power of ~1 mW. Low-temperature PL spectra were obtained at 10 K by optical cryostat (Montana Instruments) with the laser excitation wavelength of 532 nm. Circular-polarization-resolved PL measurements were performed under near-resonant excitation of 633 nm at 10 K. The circularly polarized light was generated by using a super-achromatic quarter-wave plate (Thorlabs SAQWP05M-1700) and the photo-luminescence was analyzed through the same quarter-wave plate and a linear-achromatic quarter-wave plate (Thorlabs SAQWP05M-1700) and the photo-luminescence was analyzed through the same quarter-wave plate and a linear-polarizer. We define the degree of PL circular helicity (g), which reflects the valley polarization, as \( g = \frac{[PL(\sigma^+) - PL(\sigma^-)]/[PL(\sigma^+) + PL(\sigma^-)]}{[PL(\sigma^+) + PL(\sigma^-)]} \). XPS measurements were performed using an ESCALAB 250X system (Thermo Fisher Scientific) and excited by monochromatic Al Kα radiation. Mass spectrometer (Hiden HR20) attached with temperature-programmed decomposition (TPD) was used to in-situ detect and analyze the released gas in inert atmosphere. EDS and STEM experiments were performed in FEI Titan Themis G2 300 operated at 300 kV and in Nion U-HERMES200 at 60 kV for element analysis and characterizing atomic structures of samples. Atomic-resolved AFM measurements were performed using Asylum Research Cypher in ambient atmosphere.

Fabrication and measurement of MoS2 FET device. Electron-beam lithography (EBL) is used to define the channel and the source/drain contacts with PMMA EBL resists. Metallization is implemented by thermal evaporation of 20 nm bismuth with a rate of 0.2 Å s⁻¹, followed by an Au capping layer by electron-beam evaporation (30 nm at 0.1 Å s⁻¹) at ~10⁻⁷ torr. Lift-off process is carried out in hot acetone. All electrical characterization is conducted in a vacuum environment and room temperature in a Janis probe station using a semiconductor device analyser (Agilent Technologies B1500A). The field mobilities of MoS2 were calculated according to the equation \( \mu = \frac{dI_d/dV_g}{I_d(W/C \cdot V_m)} \). In this equation, the \( L \)
Fig. 4 Growth mechanism with chalcogen monomer supply in MoX₂ (X = S, Se, Te). a Schematic diagram of adsorption, diffusion, and vacancy healing of chalcogen monomer on MoX₂ surface. The orange dotted arrows denote the motion of chalcogen monomers or dimers. b The binding energies of monomers and dimers on MoX₂ surface. The much higher energy of monomers facilitates their better adsorption on the TMD surface than dimers. c The energy profiles of vacancy healing for MoX₂ surface by using chalcogen monomers. The relatively small energy barriers of chalcogen monomer diffusion and the highly exothermic reaction at the vacancy both accelerate the self-healing of MoX₂.
Acknowledgements

This work was supported by Guangdong Major Project of Basic and Applied Basic Research with grant number 2021B0301030002 (E.W. and Kaihui Liu); the National Key R&D Program of China under grant numbers 2021YFA1400502 (M.W.) and 2021YFA1400201 (H.H.); the National Natural Science Foundation of China under grant numbers 52025023 (M.W.) and 2021YFA1400201 (H.H.); the National Key R&D Program of Chinese Academy of Sciences under grant numbers XDB33000000 (Kaihui Liu) and XDB3300200 (X.B.). We acknowledge the Electron Microscopy Laboratory in Peking University for the use of electron microscopy.

Author contributions

Kaihui Liu, F.D., X.B., and Can Liu supervised the project. Y.Z. and Can Liu conducted the sample growth and characterizations. L.D. and F.D. performed the theoretical calculations. R.Q., Q.G., and X.B. performed the STEM experiments. J.T., R.Y., and G.Z. performed the electrical measurements. Chang Liu, X.Z., and H.H. executed the low-temperature PL and valley polarization measurements. Q.W., G.X., Y.Y., J.W., Y.F., Kehai Liu, and M.W. helped sample characterizations. X.L. helped analysis electrical data. Y.Z., Can Liu, L.D., R.Q., Kaihui Liu, and F.D. wrote the article. X.B., D.Y., and E.W. revised the manuscript. All the authors discussed the results and commented on the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

Supplementary information The online version contains supplementary material available at https://doi.org/10.1038/s41467-022-28628-7.

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Peer review information Nature Communications thanks Po-Wen Chiu and the other anonymous reviewers for their contribution to the peer review of this work.

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