We report wafer-scale growth of atomically thin, three-dimensional (3D) van der Waals (vdW) semiconductor membranes. By controlling the growth kinetics in the near-equilibrium limit during metal-organic chemical vapor depositions of MoS₂ and WS₂ monolayer (ML) crystals, we have achieved conformal ML coverage on diverse 3D texture substrates, such as periodic arrays of nanoscale needles and trenches on quartz and SiO₂/Si substrates. The ML semiconductor properties, such as channel resistivity and photoluminescence, are verified to be seamlessly uniform over the 3D textures and are scalable to wafer scale. In addition, we demonstrated that these 3D films can be easily delaminated from the growth substrates to form suspended 3D semiconductor membranes. Our work suggests that vDW ML semiconductor films can be useful platforms for patchable membrane electronics with atomic precision, yet large areas, on arbitrary substrates.

INTRODUCTION

Two-dimensional (2D) transition metal dichalcogenides (TMDCs) are characterized by strong crystal anisotropy due to weak interlayer van der Waals (vdW) interactions and strong intralayer covalent bonds, resulting in vDW layered crystal structures (1, 2). Exotic physical properties, recently found in these TMDCs, are often layer-number dependent near the monolayer (ML) regime (3–8). In addition, within the unit layer, atomic-scale deformations such as point defects and grain boundaries generate distinctive 2D physical properties. For example, single point defects in WSe₂ and WS₂ MLs serve as sources for single-photon emissions (9, 10) and electronic dopants (11, 12), and mirror twin boundaries in MoSe₂ MLs provide topologically protected edge states (13, 14). To translate the local physical properties into practical device platforms, these atomic-scale deformations must be deterministically embedded in the 2D host lattices of TMDC MLs over a large area, i.e., at the wafer scale (15, 16). To this end, we report conformal growth of atomically thin MoS₂ and WS₂ films on wafer scale (including ML films) periodical 3D textured substrates, by controlled metal-organic chemical vapor deposition (MOCVD). Therein, the periodic 3D patterns include arrays of nanoscale needles fabricated on 4-inch quartz wafers and microscale trenches on 4-inch SiO₂/p⁻⁻⁻⁻Si wafers. In addition, we demonstrate the fabrication of 3D textured membranes by delaminating deposited MoS₂ and WS₂ films from the patterned substrates.

RESULTS

Periodic arrays of sharp needles (tip radius, >10 nm) on pyramidal podia were electrochemically patterned on 4-inch quartz wafers, which then served as growth substrates, as shown in Fig. 1A (see Materials and Methods). Conformal deposition of MoS₂ ML was achieved by MOCVD using Mo(CO)₆ and (C₂H₅)₂S precursors (Fig. 1B). Representative Raman scattering and photoluminescence (PL) spectra, collected from five different areas on the wafer (Fig. 1C) at room temperature, show the E₂g (386.6 cm⁻¹) and A₁g (406.4 cm⁻¹) vibration modes and the 1.88-eV light emission with the identical full width at half maximum of Δω ~ 19.8 cm⁻¹ and ΔE ~ 61 meV with similar intensities. They commonly pertain to the MoS₂ ML characteristics; layer-number-dependent PL spectra are provided for comparison (fig. S1). This conformal ML is polycrystalline, with a typical grain size of ~200 to 300 nm, as shown in sequential snapshots obtained during the growth period (Fig. 1, D to F); one can identify individual triangular facets on quartz pyramids and needles in Fig. 1E, which then merge to form continuous ML films (Fig. 1F) on the pyramid pyramids and needles (Fig. 1D). To verify the conformal coverage of the MoS₂ ML, transmission electron microscopy (TEM) specimens were prepared by a dry-cut method of the as-grown substrates (Fig. 1G), in which multiple arrays of MoS₂ ML on needles were characterized one by one by focusing individual needles located on different focal planes (e.g., in Fig. 1H, the outlined needle is in focus, but the rests are out of focus). The high-resolution TEM (HRTEM) image in Fig. 1I exhibits the conformal ML coverage over the entire needle surfaces. High-angle annular dark-field (HAADF) scanning TEM (STEM) image reveals the conformal atomic structure of the MoS₂ ML on the edge of a needle (see the magnified image and atomic model in Fig. 1J). The chemical composition of the MoS₂ ML was identified via energy dispersive x-ray spectroscopy (EDS) elemental mapping in the STEM operated at 60 kV (Fig. 1K).

We have also demonstrated the conformal deposition of MoS₂ ML on SiO₂/p⁻⁻⁻⁻Si trench substrates, which were periodically patterned with various pitch distances at the micrometer scale (Fig. 2, A and B); the square patterns of different colors shown in the optical microscopy (OM) image in Fig. 2A are due to the different pitch distances (fig. S2). The cross-sectional HAADF-STEM images directly confirm the conformal deposition of MoS₂ ML on these trenches (Fig. 2C). Similar to the growth on quartz pyramids and needles, polycrystalline texturing was achieved; discrete ML crystal facets (~200 to 300 nm in size) were conformally grown on the trenches (Fig. 2D) to form continuous ML films (Fig. 2, E and F). PL mapping images (Fig. 2G), focused on either the top or bottom of the trenches, show a uniform
PL intensity distribution for the MoS$_2$ MLs, suggesting uniform and conformal ML coverage over the 3D trenches. To verify a reliable ML channel connection along with the trench coverage, we patterned Au electrodes over various channel lengths by electron beam (e-beam) lithography/lift-off and fabricated field-effect transistor (FET) devices (Fig. 2H and fig. S3) across multiple trenches. Using the transfer length method, we confirmed that continuous and uniform channels were formed over multiple trenches, where the channel resistance was found to be linearly proportional to the length; see also fig. S4 for results at the millimeter length scale. By direct comparison with FETs formed on flat ML without trenches, we have also extracted the sheet resistance ($R_s$) to be 6.45 megohm, which is slightly higher than 4.88 megohm at the flat ML regions; see also fig. S5. Accordingly, we measured the contact resistance ($R_c$) as 24 kilohm, which is also slightly higher than 226 kilohm at the flat ML regions (Fig. 2I). The higher channel resistance can be attributed to complex grain boundaries over the trench edges, as further discussed below. The transfer curves ($V_g$-dependent $I_d$ at $V_d = 0.1$ V) show the similar n-type channel characteristics with a lower FET mobility of 3.0 cm$^2$ V$^{-1}$ s$^{-1}$ compared with 4.5 cm$^2$ V$^{-1}$ s$^{-1}$ for flat ML regions (Fig. 2J).

Using W(CO)$_6$ and (C$_2$H$_5$)$_2$S precursors, we have also achieved the conformal growth of WS$_2$ MLs on an array of sharp SiO$_2$/p+-Si needles patterned on a 4-inch wafer-scale quartz needle array. The ML uniformity of WS$_2$ regarding its semiconductor properties was verified via PL mapping across the ML tip (1 µm in height) arrays, as shown in the OM image in Fig. 3A. The PL intensity was regularly enhanced at the needles, and the periodicity matched the needle pitch (Fig. 3B); see fig. S6 for a comparison with results from ML TMDC films on planar substrates. At the ML tips, the PL intensity is enhanced by about five times compared with that at the flat region (Fig. 3C and D) because of the geometrically enhanced optical field at the needle curvature; see Fig. 3E and fig. S7 for similar Raman intensity variations. In any case, both the characteristic PL and Raman peaks remain spectroscopically identical regardless of the position within the 3D ML film, with the consistent peak energies (1.99 eV and 353 cm$^{-1}$) and the full width at half maximum (65 to 68 meV and 13.4 cm$^{-1}$), confirming the ML uniformity; see fig. S8 for similar growth characteristics of 3D MoS$_2$ films and fig. S9 for the second harmonic generation experiments. These conformally continuous 3D WS$_2$ films can be easily delaminated from the growth substrates to form suspended membranes, allowing them to float in deionized (DI) water, because of the weak vdW interaction with the quartz substrates (Fig. 3F); see fig. S10 and movie S1 for the delamination process. It turned out that the ML membranes inevitably collapsed at the needle regions during drying of the DI water on the TEM grids presumably because of a finite capillary force at the sharp needle curvature (fig. S11). Nevertheless, it is
remarkable to observe that the few-layer films robustly survived, as shown by in-plane TEM images, where the pyramidal and needle shapes were intact in the delaminated membranes (Fig. 3G); see also movie S2. We observed different selected-area electron diffraction (SAED) patterns (Fig. 3H) from the planar (yellow) and pyramidal (red) regions, which can be attributed to different diffraction paths of e-beams, associated with the different crystal angles with respect to the base plane of the substrate (Fig. 3I); see fig. S12 for detailed analyses of the electron diffraction pattern observed on the needles. We acquired PL spectra for the MoS2 and WS2 films, including ML films, before and after delamination and observed nearly identical spectral features, except for the slight blue shift of 20 to 30 meV (fig. S13). The Raman spectra were also identical. These observations suggest that the suspended membranes preserve their original crystal textures upon delamination. Clearly, these atomically thin membranes are conformally continuous at the sharp needle tips, as well as in the side (flatter) regions (Fig. 3, J to M). We found that the polycrystalline textures are maintained with the characteristic grain boundaries, particularly at the tip curvature (radius of ~30 nm). We confirmed that identically thick WS2 layers (7.8 nm; 13 layers) were conformally deposited on the needles, regardless of the aspect ratio, to form continuous 3D films.

DISCUSSIONS

The vdW ML epitaxy is characterized by a weak interaction of the deposited MLs with the target substrate; thus, in principle, ML crystal growth on a flat 2D substrate is not strictly affected by local variations on the substrates, such as morphologies, crystal texture, and strain (17). Our 3D ML growth proceeded in the near-equilibrium regime (18). As we observed stable crystal facets shown in Fig. 4A, the growth is dictated by surface energy minimization during crystallization, followed by subsequent edge growth to form continuous ML films (19, 20). Then, to kinetically accommodate this ML crystallization over periodic 3D textures at the nanometer scale, i.e., lateral 3D textured ML crystallization, we maintained a slow lateral growth rate of 0.15 nm/min by controlling the flow rate of MO sources with low partial pressures ($p_{MoCO}_6 \sim 10^{-3}$ torr) during MOCVD for MoS2 and WS2 ML (Fig. 4, B to D); see the Supplementary Materials for a growth rate estimation. Conventional powder CVD growth (Fig. 4E) (21), in which substantially higher precursor vapor pressure ($p_{MoO}_3 \sim 10^{-2}$ torr) is usually generated from solid precursors, produces a much higher growth rate (22), i.e., ~1500 nm/min in our case, and resulted in non-uniform coverage on periodic 3D textures (Fig. 4, F to H, and fig. S14). The primary process variables defining the conformal growth characteristics include the partial pressure of precursors and the resultant growth rates, determining the sticking coefficient, β, as a key parameter (23, 24). In thin film deposition, conformality depends on the reactive sticking probability of adatoms, where β is defined as the ratio of the number of adsorbate atoms (flux sticking) to atoms that impinge upon the surface (incident flux). Thus, the lower β values enable deeper diffusion of the adatoms into the 3D features, leading to better conformality. Specifically, β can be expressed as:

$$\beta = \frac{\text{flux sticking}}{\text{incident flux}} = \frac{v/p}{v/\sqrt{2mnkT}},$$

where $v$ is the lateral growth rate (nm/min),
Fig. 3. Optical properties of 3D TMDC ML films on a needle array and their membranes by delamination from 3D textured substrates. (A) OM image of a conformally coated WS₂ ML on a SiO₂/p⁺-Si needle array. Inset: Magnified SEM image of the substrate. (B) Large-area PL image at the WS₂ exciton peak (hv = 1.99 eV) obtained from the region corresponding to (A). (C) Magnified PL image. (D) PL and (E) Raman spectra obtained from the needle region (red and blue) and flat region (black). Inset: PL intensity line profile indicated in (C). (F) Photographs of the peel-off process and illustration of a delaminated vdW WS₂ membrane separated from the 3D substrate. Photo credit: Gangtae Jin, Pohang University of Science and Technology. (G) Low-magnification TEM image of a few-layer 3D WS₂ membrane at a −36.2° tilt angle. (H) SAED pattern of the 3D WS₂ membrane. (I) Diffracted beam path for planar and tilted TMDC crystals on the pyramidal array. (J) Low-magnification TEM image of the suspended WS₂ needle array at the 36.2° tilt angle. (K) Low-magnification TEM image of sharp 13-L WS₂ needle at a tilt angle of approximately 90°. (L and M) High-magnification TEM images of the (L) tip region (red) and (M) side region (yellow) of the WS₂ needle.

Fig. 4. Kinetic origin of conformal ML deposition via MOCVD process. (A) TEM images of MOCVD-grown MoS₂ crystals for different growth time. (B) False-color dark-field (DF) TEM image of suspended MOCVD-grown MoS₂ ML film on holey carbon grid. (C) Grain size distribution of MOCVD MoS₂ crystals. (D) Schematic of 3D ML texturing by MOCVD. (E) OM images of powder CVD-grown MoS₂ crystals for different growth time. (F) DF-TEM image of powder CVD-grown MoS₂ MLs. Inset: SAED pattern from MoS₂ ML film. (G) Grain size distribution of powder CVD-grown MoS₂. (H) Schematic of CVD-grown MLs from solid powder precursors.
The growth of 4-inch wafer-scale ML MoS₂ by MOCVD

Growth of 4-inch wafer-scale ML MoS₂ by MOCVD

The growth of 4-inch wafer-scale ML MoS₂ was achieved using high-purity gas precursors, Mo(CO)₆ (99.99%; Sigma-Aldrich) and (CH₃)₂S (98%; Sigma-Aldrich). Four-inch quartz and SiO₂/p+-Si wafers were placed in the center of a 6-inch hot-walled quartz tube furnace. Before the MOCVD process, the furnace was purged for 1 hour to eliminate residual contaminants, and the temperature was ramped up to 535°C for 30 min. The growth proceeded for 26 hours with partial pressure of precursors of 4.2×10⁻⁵ torr for Mo(CO)₆ and 10⁻² torr for (CH₃)₂S. The base pressure of the reactor was ~7 torr under the carrier gas flow of 150 standard cubic centimeters per minute (sccm) for Ar (99.9999%) and 1 sccm for H₂ (99.9999%).

Water-assisted transfer of 3D TMDC membranes

Delamination and transfer of TMDC membranes can be conducted without the typical spin-coating process of polymer layers (poly(methyl methacrylate) and polystyrene) and removers (acetone and toluene). The outer edges of pristine TMDC films grown on 3D textured substrates were gently carved with a knife. The newly exposed SiO₂ surface was dipped into DI water, and the water penetrated the interface between the TMDCs and SiO₂ because of their difference in wettability. The entire 3D TMDC membrane, floating on water, was picked up onto an arbitrary substrate. Last, the transferred 3D TMDC membranes were naturally dried under ambient conditions.

SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/5/7/eaaw3180/DC1

Fig. S1. Layer-number-dependent PL spectra (ML and bilayer) of MoS₂ and WS₂ films.
Fig. S2. OM images as different pitch sizes in patterned SiO₂/p⁺-Si trenches.
Fig. S3. False-color SEM image of the FET devices on flat regions.
Fig. S4. Length-dependent resistance of 3D MoS₂ ML films on microtrench substrates.
Fig. S5. PL intensity maps and the corresponding PL spectra of TMDC ML films on planar SiO₂/p⁺-Si substrates.
Fig. S6. PL intensity maps and the corresponding PL spectra of TMDC ML films on planar SiO₂/p⁺-Si substrates.
Fig. S7. Raman mapping of 3D WS₂ ML films on SiO₂/p⁺-Si needle arrays.
Fig. S8. Raman and PL mapping of 3D MoS₂ ML films on SiO₂/p⁺-Si needle arrays.
Fig. S9. Second harmonic generation in MoS₂.
Fig. S10. Schematic illustration of the DI water-assisted transfer of 3D TMDC membranes.
Fig. S11. TEM images of delaminated WS₂ ML films on a TEM grid.
Fig. S12. Local diffraction patterns generated on the 3D WS₂ membranes.
Fig. S13. PL and Raman spectra of MoS₂ ML and WS₂ ML films before and after delamination.
Fig. S14. Nonconformal MoS₂ film growth on microtrench substrates by a powder CVD method.
Fig. S15. MOCD growth of MoS₂ films on 3D trench substrates at a faster growth rate.

Section S1. Pyramid structure analysis using the diffraction patterns
Section S2. Determination of sticking coefficients
Section S3. Powder CVD growth of MoS₂ ML crystals

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Movie S1. Peeling off the WS₂ ML films from the quartz substrates by immersing in DI water.

Movie S2. TEM tilting of suspended 3D WS₃ membranes.

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