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Chemically Engineered Substrates for Patternable Growth of Two-Dimensional Chalcogenide Crystals

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ABSTRACT: The key challenge of direct integration of two-dimensional (2D) chalcogenide crystals into functional modules lies on precise control of the nucleation sites of the building blocks. Herein, we exploit the chemical activities and surface engineering of the substrates to manipulate the nucleation energy barrier of 2D crystals, and thereby realize the patternable growth of 2D crystals. The selective-region chemical modifications of the substrates are achieved via micro-contact printing combined with the elegant self-assembly of octadecyltrichlorosilane (OTS) molecules on the substrates. The patternable growth method is versatile, and can be used as a general strategy for growing a broad class of high-quality 2D chalcogenide crystals with tailorable configurations on a variety of chemically engineered substrates. Moreover, we demonstrate flexible transparent electrodes based on large-scale patterned nanogrids of topological insulator Bi$_2$Se$_3$, which possess tailored tradeoff between electric conductivity and optical transmittance across the visible to near-infrared regime. We hope this method may open an avenue to the efficient integration and batch-production of 2D chalcogenide crystals, and could inspire ongoing efforts of the fabrication of van der Waals heterostructures.

KEYWORDS: patternable growth, 2D crystals, chemical activity, substrate
Since the discovery of graphene, the realm of two-dimensional (2D) chalcogenide crystals hosting rich electronic or optoelectronic properties is intensely explored.\textsuperscript{1-3} To achieve the full potential of 2D chalcogenide crystals, it is of technological significance to integrate the 2D nanostructures into sophisticated functional electronic or optoelectronic modules with patterned architectures, for example, the integrated circuits (IC). Actually, this bottom-up paradigm is persistently pursued since the inception of nanoscience and nanotechnology, \textit{e.g.} oriented carbon nanotubes\textsuperscript{4, 5}, semiconductor nanowires arrays,\textsuperscript{6, 7} inorganic nanocrystal arrays\textsuperscript{8} and organic semiconductor arrays\textsuperscript{9, 10}. To this end, two approaches are usually employed. One approach is the post-growth manipulation of the random nanoscale building blocks,\textsuperscript{7, 11} while the other one is the direct configuration of the nanoscale building blocks into predesigned pattern during growth,\textsuperscript{9, 10} \textit{viz.} the patternable growth of the nanoscale building blocks. The latter approach is generally believed to be superior for its simple procedure, and more importantly, it avoids potential damages or contaminations of the vulnerable nanoscale materials. With regard to the patternable growth of the nanoscale building blocks, the key point is the precise control over nucleation sites and the concomitant layout of patterned crystals, including the size, geometry and orientation, \textit{etc.}

In terms of the controlled growth of various 2D crystals, van der Waals epitaxy was proved to be a versatile technique on substrates like mica and SiO$_2$/Si, which is tolerant of considerable lattice mismatch.\textsuperscript{12-18} The thickness and nucleation density can be well controlled by tuning the growth parameters such as the deposition temperature, pressure and growth duration. However, the tuning of these parameters still falls short of precisely controlling the nucleation sites therewith usually random locations of the resultant 2D crystals, let alone the patternable growth. From the perspective of the crystal nucleation and growth mechanism, the rationale dominating the patternable growth of the building block is to induce a dramatic energy difference of nucleation barrier. Therefore, the current methodology is concentrated on the selective-region engineering of the substrates. Nevertheless, the finite methods reported for the patternable growth of 2D crystals thus far are either short of scalability and freedoms of pattern design or plagued by substrates. For instance, the mask-assisted plasma etching performs physical surgeries of the substrate and suffers from scalability.\textsuperscript{13, 17, 18} The seeded growth method heavily rely on costly and ponderous micro-fabrication technologies.\textsuperscript{19-21} Moreover, the patterned architectures show poor crystallinity and limited freedoms of pattern design. The microintaglio printing method demonstrates incredibly flexible freedoms of pattern design and distinct scalability.\textsuperscript{22} However, the microintaglio printing van der Waals method is plagued by substrates due to its patternable growth process. Clearly, versatile and scalable patternable growth methods for high-quality 2D crystals are highly desired. Herein, we exploit the chemical activities of the substrates to achieve large-area patternable growth of 2D chalcogenide crystals. Our method not only shows flexible
freedoms of pattern design and excellent scalability, but also remarkably lifts up the limitations imposed by substrates due to its growth mechanism, which has not been reported for the patternable growth of 2D crystals.

**Figure 1.** Patternable growth of 2D layered crystals. (a) Schematic illustration of the procedure of patterning 2D crystal arrays by chemically engineering of the substrates. (b) Layered crystal structure of Bi$_2$Se$_3$, Bi$_2$Te$_3$ and In$_2$Se$_3$ composed of QLs. (c) Typical optical microscope (OM) image of large-area patterned Ti Bi$_2$Se$_3$ 2D nanoplate arrays on mica. Inset: zoom-in OM image of (c), scale bar is 20 µm. (d) AFM image of two adjacent patterned 2D Bi$_2$Se$_3$ nanoplates on mica. (e) Typical Raman spectrum of the patterned 2D Bi$_2$Se$_3$ nanoplates. (f) Raman mapping ($E_g^2$ mode) of two adjacent patterned 2D Bi$_2$Se$_3$ nanoplates on mica.

**RESULTS AND DISCUSSION**

The key of this strategy relies on the manipulation of the nucleation energy barrier by selective-region chemical modification of the substrates. As schematically illustrated in Figure 1a, we take advantage of the micro-contact printing technique combined with the molecular self-assembly of octadecyltrichlorosilane (OTS) molecules on muscovite mica (KAl$_2$(Si$_3$Al)O$_{10}$(OH)$_2$), which is ultra-hydrophilic (Figure S2a) and atomically flat (Figure S2d). As elastomeric polydimethylsiloxane (PDMS) stamp conformally contacted with mica substrate, OTS molecules were printed onto the substrate. And thereby OTS self-assembled monolayers (OTS-SAMs) form on the surface via chemical bonds, perfectly reproducing the artificially tailorurable structures of the stamp (Figure S1). Contact angle examinations (Figure S2b), scanning electron microscope (SEM) (Figure S2e), atomic force
microscope (AFM) (Figure S2f) testify that mica was readily modified by OTS-SAMs and the pattern transfer proceeded with excellent fidelity.

Bi$_2$Se$_3$, Bi$_2$Te$_3$ and In$_2$Se$_3$ are studied in this work as the model systems to verify the efficacy of this methodology for the patternable growth of 2D crystals, because of their potential in electronics or optoelectronics. In detail, Bi$_2$Se$_3$ and Bi$_2$Te$_3$ are members of the family identified to be topological insulators (TIs), a quantum phase matter combining trivial gaped bulk states with nontrivial surface/edge states.$^{24,25}$ This exotic electronic signature renders TIs to be promising building block candidates for next-generation low-power consumption electronics. In$_2$Se$_3$, a traditional phase change material, has recently been proved to be highly efficient in photocurrent conversion.$^{13,22,26}$

As depicted in Figure 1b, the layered chalcogenides of Bi$_2$Se$_3$, Bi$_2$Te$_3$ and In$_2$Se$_3$ have similar crystal structures, composed of planar quintuple layer (QL) units. Atoms within each QL unit (height ca. 1 nm) are strongly bonded by covalence while adjacent QLs are weakly connected by van der Waals interactions. The as-modified mica substrates together with the Bi$_2$Se$_3$ crystal powders were loaded into the apparatus similar to previous works (Figure S3).$^{13,14,17,18,22,27}$ Strikingly, preferential crystallization of Bi$_2$Se$_3$ nanoplate arrays takes place on the modified regions of the substrate unambiguously. Figure 1c demonstrates a typical optical microscope (OM) image of a large-area patterned Bi$_2$Se$_3$ nanoplate arrays. The arrays correspond to 10 µm square domains and 5 µm intervals, corresponding to the density of nanoplate arrays as high as 2.8 × 10$^6$ per square inch. The uniform color contrast is indicative of the uniform thickness of the nanoplate arrays. Such uniformity was further confirmed by AFM and micro-Raman spectroscopy. The thickness of the nanoplates can be well controlled down to 4 nm (4 QLs) with excellent flatness (Figure 1d). Raman spectrum exhibits three sharp peaks, characteristic of Bi$_2$Se$_3$ (Figure 1e).$^{14,28}$ Raman mapping also reveals the ultrahigh homogeneity of nanoplate arrays as well as the inappreciable crosstalk between the crystals (Figure 1f).

To check the versatility of this methodology, we change the source crystals, stamp relief structures as well as the target substrates. Figure S4e presents the optical images of as-grown In$_2$Se$_3$ nanoplate arrays using PDMS stamp identical to that of Bi$_2$Se$_3$ nanoplate arrays. By utilizing PDMS stamp with reverse relief structures (Figure S1d), large-area Bi$_2$Se$_3$ nanogrids were achieved (Figure 4b, Figure S4b-d). Furthermore, other configurations, including different domain sizes (Figure S5) and elaborate geometries of the patterned crystal were achieved (Figure S6). These observations further corroborate that the configurations of the patterned nanostructures are purely contingent on the relief structure of PDMS stamp or the manipulation of the PDMS stamp, justifying the highly flexible freedoms of pattern design.
Figure 2. Structural analyses of the patterned Bi$_2$Se$_3$ nanoplate arrays. (a) XRD pattern of Bi$_2$Se$_3$ nanoplate arrays on mica (red line), freshly cleaved muscovite mica (blue line) and reference diffractogram of rhombohedral Bi$_2$Se$_3$ (black line, PDF#33-0214). Note that the intensity is in logarithmic scale to manifest the signals of Bi$_2$Se$_3$. (b) Low-magnification bright field TEM image of Bi$_2$Se$_3$ nanoplate arrays. (c) HRTEM image of Bi$_2$Se$_3$ nanoplate edge. (d-j) SAED patterns corresponding to the sites 1-7 in (b). Scale bar: 5 nm$^{-1}$ (k) Histogram of the SAED angle of 20 different sites.

Subsequently, to evaluate the quality of the patterned 2D crystal arrays, we carried out systematic structural characterizations, including the X-ray diffraction (XRD), transmission electron microscope (TEM), selective area electron diffraction (SAED) and energy-dispersive spectroscopy (EDX). XRD measurements of the as-synthesized thick Bi$_2$Se$_3$ nanoplate arrays were directly performed on mica without transfer. The as-achieved Bi$_2$Se$_3$ crystal arrays crystallize into the phase-pure rhombohedral structure, as revealed by the XRD patterns (Figure 2a). Meanwhile, the peaks are well indexed into the (0001) family planes of Bi$_2$Se$_3$ rhombohedral crystal, indicating that the c-axis of as-grown crystal is perpendicular to the mica plane. Moreover, the patterned Bi$_2$Se$_3$ 2D crystal arrays were faithfully transferred onto the holey carbon film with the aid of polymethyl methacrylate (PMMA) for TEM characterizations (Figure 2b and Fig S7a). The EDX analysis confirms the atomic ratio of Bi and Se approximately to be 2 to 3 (Figures S7b, c). As shown in the high-resolution TEM
image, the Bi₂Se₃ crystals are of high-quality with a clear lattice spacing of ~0.21 nm, well consistent with the lattice plane of (11−20) in rhombohedral Bi₂Se₃ (Figure 2c). Interestingly, as manifested in Figure 2d-j, the SAED patterns of adjacent nanoplates show almost identical lattice direction (deviation smaller than ±1°, Figure 2k), reminiscent of the single-crystalline nature of as-patterned Bi₂Se₃ crystal arrays as well as the substrate-induced orientation alignment on unmodified mica.¹³,¹⁷ The single-crystalline nature can be further confirmed by the perfect hexagonal diffraction pattern and same orientation of the thick Bi₂Se₃ nanoplate arrays (Figure S9c). In brief, the systematic structural characterizations indicate the high quality of as-patterned Bi₂Se₃ nanoplate arrays obtained via our method.

**Figure 3.** Patternable growth mechanism. (a) AFM image of the pristine OTS-SAMs on mica. Scale bar: 5 µm. (b, c) AFM image of the OTS-SAMs on mica after annealing at 490 °C and 750 °C, respectively. Scale bar: 5 µm. (d, e, f) OM images showing the morphology evolution of Bi₂Se₃ nanoplate arrays during the patternable growth with mild rate of carrier gas (typically <150 standard cubic centimeter per minute (sccm)). Scale bar: 10 µm. Insets in (d, e) are AFM images of one patterned Bi₂Se₃ nanoplate at the intermediate state of nucleation and during growth, respectively. Scale bars: 5 µm. (g) OM images of patterned Bi₂Se₃ nanoplate arrays on SiO₂/Si. (h) Number of nuclei per domain plotted against the area of domain with violent rate of carrier gas (typically >500 sccm). Insets are OM images of nuclei on
mica corresponding to respective domain size. (i) SEM images of patterned Bi$_2$Se$_3$ nanoplate arrays with domain size ~2 µm. Scale bar: 5 µm.

For better manipulation of the patternable growth, it is of prime significance to investigate the underlying mechanism. Consequently, we conducted control experiments as follows: chemically modified substrates were annealed under conditions similar to that of general growth, whereas no source powders were loaded. The pristine OTS-SAMs are densely compact and uniform in height, as evident in the AFM image (Figure 3a and Figures S8a, d). In contrast, OTS-SAMs turn into loose and discrete particles with same height in the modified domain after annealing at 490 °C (Figure 3b and Figures S8b, e). This phenomenon becomes more self-evident when the annealing temperature is elevated to 750 °C (Figure 3c and Figure S8c, f). Thus, we postulate that these particles serve as the nucleation sites, in good consistency with the crystal growth theory dictating that heterogeneous nucleations need dramatically lower nucleation energy barrier. Meanwhile, the general morphology evolution of Bi$_2$Se$_3$ nanoplate arrays during growth is clearly distinguished and presented in Figures 3d-f. The snapshot captured at the intermediate state of nucleation shows that uniform discrete nuclei about 2 QLs (~2 nm) form within the modified domain (inset of Figure 3d). When the nuclei fully coalesce, as shown in Figure 3e, the growth proceeds in a layer-by-layer growth mode, a well-established signature of 2D crystal growth (inset of Figure 3e). These observations further validate our postulation that discrete particles serve as the nucleation centers. Furthermore, the subsequent layer-by-layer growth scenario facilitates the thickness control of the as-patterned 2D layered crystals, simply by tuning the growth duration (Figure S9). Notably, the thick patterned Bi$_2$Se$_3$ architectures typically exhibit approximately hexagonal morphology (Figure 3f and Figure S9c), deviating from the predesigned square relief structures. This phenomenon is putatively due to the anisotropic growth of Bi$_2$Se$_3$, which is in turn determined by its rhombohedral crystal lattice.

Moreover, it is worth noting that this method is also applicable to other substrates. For example, nanoplate arrays of chalcogenide crystals can form on modified SiO$_2$/Si and glass with considerably good crystallinity (Figure 3g and Figure S10). In particular, the 2D chalcogenide crystals are usually not aligned on SiO$_2$/Si because of the amorphous property of SiO$_2$. However, the patterned Bi$_2$Se$_3$ nanoplate arrays are aligned in our method, as clearly demonstrated in Figure 3g. The trick is presumably due to the growth mechanism, in which the coalesced uniform nuclei (~2 QLs) may serve as the buffer layer and therefore lifts up the limitations imposed by substrates. The hypothesis is supported by the well-developed buffer-layer methodology, which has been verified to be powerful for both traditional epitaxy and van der Waals epitaxy in removing the limitations imposed by the substrate. For traditional epitaxy, the most successful story is the growth of single-crystalline GaN epifilms via buffer-layer mediation, which enables the revolution of blue light diodes.
Markedly analogous to our case, it is claimed that high-quality $\text{Bi}_2\text{Se}_3$ epifilms can be achieved via van der Waals epitaxy on the buffer layer of $\text{Bi}_2\text{Se}_3$ itself, which also effectively lift up the limitations imposed by substrates.\textsuperscript{16, 32} So all the findings coherently confirm that our patternable growth method could lift up the limitations imposed by substrates. Furthermore, these insulating substrates are compatible with modern electronics technologies.

In addition, we note that the window for patternable growth is relatively narrow: the substrate temperature and the flow rate of carrier gas are identified to be critical parameters to achieve the patternable growth of 2D crystals. Low substrate temperature is more favorable to the formation of continuous films on both the modified and unmodified substrate regions, rather than discrete nanoplate arrays merely on modified substrates with minimal in-between crosstalks (Figures S11). The result can be elucidated by classic film growth theory. According to the film growth theory, the nucleation rate is co-determined by pressure, substrate temperature and nucleation energy, etc (Figures S11g).\textsuperscript{33} Because the specific substrate is always exposed to same ambience, it is therefore impossible to achieve patternable growth by tuning pressure $P$ and substrate temperature $T$. But extreme conditions of $P$ or $T$ can result in ultrafast or ultraslow nucleation rate, which is destructive for patternable growth. In this case, low substrate temperature leads to ultrafast nucleation rate, and thereby continuous films tend to form. Meanwhile, a higher flow of carrier gas would result in the difficulty for the coalescence of each nuclei into one single uniform epilayer within one modified domain. Discrete nanoplates tend to form within one modified domain (insets of Figure 3h). Moreover, the number of nuclei within one modified domain are positively correlated to the domain area, and single nucleus was achieved by diminishing the domain size down to $\sim$2 $\mu$m (Figure 3h, i and Figure S12). This observation is in close analogy to the patternable growth of non-layered inorganic nanocrystal arrays\textsuperscript{8} or organic semiconductor arrays\textsuperscript{9} by means of OTS-SAMs. The contrast highlights the layered structures of 2D crystals.
Continuous nanosheets of topological insulator Bi$_2$Se$_3$ have been recognized by our group and others as a promising candidate to flexible transparent electrodes, which are immune to surface contaminations and bending.\textsuperscript{34,36} Compared to ITO electrodes, Bi$_2$Se$_3$ electrode possesses extraordinary transmittance in the near-infrared regime, and relatively lower transmittance in the visible light regime. In this work, we demonstrate the patterned large-area Bi$_2$Se$_3$ nanogrids based flexible transparent electrodes (Figure 4a, b), which shows excellent optical transmittance across the visible to near-infrared regime with good electric conductivity. In comparison with ITO electrode, the patterned Bi$_2$Se$_3$ nanogrids have comparable transmittance in the visible light regime, and superior transmittance in the near-infrared regime (Figure 4c). The excellent optical transparency is attributed to twofold reasons: one is due to its grid structure; the other one is the exotic spin nondegenerate surface states of topological insulator, which prohibits the carriers of surface states from the direct photoexcitation.\textsuperscript{34}

The sheet resistances were measured on the large-area patterned Bi$_2$Se$_3$ nanogrids in a four-probe configuration. As presented in Figure 4d, the sheet resistance of
Bi$_2$Se$_3$ nanogrids is larger than that of its continuous film counterpart, because the “filling factor” of nanogrids is always less than 1 (In our case as indicated in Figure 4b, the “filling factor” is ca. 55.56%).$^{35}$ The sheet resistance of Bi$_2$Se$_3$ nanogrids can be tuned by its thickness. Obviously, for specific applications, the desired tradeoff between the optical transparency and sheet resistance can, in principle, be tuned by tailoring the grid structures (“filling factor”) and thickness of nanogrids. The preliminary results suggest that our patternable growth method is versatile and easy to scale up for batch production of topological insulator Bi$_2$Se$_3$ nanogrids based flexible transparent electrodes for potential use in display windows, touch screens and wearable electronics.

CONCLUSION

In summary, we exploit the chemical activities of the substrates and develop a scalable and versatile method for the patternable growth of 2D chalcogenide crystals. Remarkably, our method could lift up the limitations imposed by substrates. Our findings may open an avenue to the integration and batch production of 2D crystal devices and inspire ongoing efforts dedicated to the fabrication of the in-plane or vertical van der Waals heterostructures.$^{15,37}$

EXPERIMENTAL SECTION

Chemical modification of the substrates.

The procedure of the fabrication of PDMS stamp was same as that in our previous work.$^{22}$ For the chemical modifications of the substrates, OTS/n-hexane solution (0.5-1 mmol/L) solution was firstly applied onto PDMS stamp using a cotton swab for several times. It could be seen that n-hexane evaporated very quickly. Then the PDMS stamp conformally contacted with the substrates for 10-30 minutes. Lastly, PDMS stamp was peeled off from the modified substrates with care. N-octadecyltrichlorosilane (purity 95%, ~ 5-10% branched isomers) was purchased from Alfa Aesar, stored in glove box in Argon ambience and used as received.

Patternable growth of 2D materials.

The source powders of Bi$_2$Se$_3$ (purity 99.999%), Bi$_2$Te$_3$ (purity 99.999%) and In$_2$Se$_3$ (purity 99.99%) were purchased from Alfa Aesar and used as received. The source powders together with chemically modified substrates were loaded into horizontal tube furnace (Lindberg/Blue M) with 1-inch diameter quartz tube heated by a 12-inch long. The substrates were typically put sequentially from 6 cm to 13 cm away from the heat center of the furnace. The carrier gas was argon (Ar). Before elevating temperature, the system was thoroughly purge with Ar several times. Typically, the temperature was elevated to 470-500 °C for the patternable growth of Bi$_2$Se$_3$ and
Bi$_2$Te$_3$ and 720-750 °C for the patternable growth of In$_2$Se$_3$. The elevation rate of temperature was generally set ~30 °C per minute and growth duration is tuned from 5 to 60 min.

**Characterization.**

The OTS self-assembled monolayer and patterned 2D chalcogenide crystals (Bi$_2$Se$_3$, Bi$_2$Te$_3$, and In$_2$Se$_3$) were characterized by optical microscopy (Olympus DX51 microscope), scanning electron microscopy (Hitachi S-4800, acceleration voltage 0.5-2 kV), atomic force microscopy (Vecco Nanoscope IIIa and Bruker Dimension Icon, tapping mode). Transmission electron microscopy (TEM) were performed on FEI Tecnai F30 (acceleration voltage 300 kV) equipped with an energy dispersive X-ray spectrometer (EDX). X-ray diffraction spectroscopy (XRD) was performed on Bruker D8 Advanced diffractometer in modified Bebye-Scherrer geometry with Cu Kα1 radiation. Raman spectra and mapping were conducted on a Horiba HR800 Raman system using 514 nm laser for excitation. Contact angle tests were measured on OCA 20. UV-Vis-IR transmittance measurements were performed on Perkin Elmer Lambda 950 spectrophotometer. Sheet resistances were measured on Res Map 178 (Creative Design Engineering Inc., Silicon Valley).

**ASSOCIATED CONTENT**

**Supporting Information**

Supplementary Text, Supplementary Figures S1-S12, and additional References. This material is available free of charge via the Internet at http://pubs.acs.org.

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**Notes**

The authors declare no competing financial interests.

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