Probing the crystallographic orientation of two-dimensional atomic crystals with supramolecular self-assembly

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Probing the crystallographic orientation of two-dimensional (2D) materials is essential to understand and engineer their properties. However, the nondestructive identification of the lattice orientations of various 2D materials remains a challenge due to their very thin nature. Here, we identify the crystallographic structures of various 2D atomic crystals using molecules as probes by utilizing orientation-dependent molecule-substrate interactions. We discover that the periodic atomic packing of 2D materials guides oleamide molecules to assemble into quasi-one-dimensional nanoribbons with specific alignments which precisely indicate the lattice orientations of the underlying materials. Using oleamide molecules as probes, we successfully identify the crystallographic orientations of ~12 different 2D materials without degrading their intrinsic properties. Our findings allow for the nondestructive identification of the lattice structure of various 2D atomic crystals and shed light on the functionalization of these 2D materials with supramolecular assembly.
Two-dimensional (2D) atomic crystals, endowed with unique ultrathin planar and well-ordered atomic structures, have shown a broad range of attractive properties, which are dramatically different from their bulk counterparts. Among these properties, electrical transport, thermal conductance, piezoelectric characteristics, optical properties and so on are significantly dependent on their 2D crystallographic orientations, especially for those with in-plane anisotropic structures such as black phosphorus and ReS$_2$. Besides these intrinsic properties, modulating the properties of 2D atomic crystals by strain, stacking and size-confinement is also highly dependent on their pristine lattice orientations as indicated by various theoretical and experimental studies. Therefore, probing the crystallographic orientation of these materials is essential for understanding and controllably tuning their properties in an expanding array of 2D atomic materials. The most straightforward characterization is to directly image the atomic structure of 2D materials with scanning transmission electron microscope (STEM) or scanning tunneling microscope (STM). However, the acquisition of atomically resolved images by both STEM and STM has critical requirements on sample preparation, equipment and imaging skills, which makes these approaches very expensive and low throughput. More importantly, the imaged samples are located on specialized substrates such as transmission electron microscope grids or conducting substrates, which hinders their further applications. Recently, orientation-dependent intensities of second harmonic generation (SHG) and polarized Raman spectroscopy have been observed in several 2D atomic crystals, providing an alternative approach for identifying lattice orientation of several 2D materials, such as ordered-layered BN$_2$, MoS$_2$, MoSe$_2$, MoTe$_2$, WS$_2$, WSe$_2$, NbSe$_2$, TaS$_2$ and so on (Fig. 1b–g and Supplementary Fig. 2), and these nanoribbons aligned along three directions with rotations of ~120° (Supplementary Fig. 3) as schematically illustrated in Fig. 1a over a large area (Supplementary Figs. 4 and 5). In addition to these hexagonal crystals, nanoribbons on black phosphorus with tetragonal symmetry and ReS$_2$ with distorted 1T structure also displayed specific orientations along two and one directions, respectively (Fig. 1h, i). We ruled out the possible formation of oleamide nanoribbons in solution through dynamic light scattering (DLS) measurements (Supplementary Fig. 6) and a control experiment on the self-assembly of 5, 10, 15, 20-tetraphenyl-21H, 23H-porphine cobalt (II) (CoTPP) on MoS$_2$ (Supplementary Figs. 6 and 7), and therefore, confirmed that oleamide nanoribbons were formed on the surface of 2D materials instead of in solution. The dramatically different orientations of oleamide nanoribbons on 2D atomic crystals with varied crystallographic structures suggest that the alignment of nanoribbons is very likely to be guided by the atomic structures of the underlying substrates.

Orientation-correlations between oleamide nanoribbons and 2D materials. To correlate the orientations of the oleamide nanoribbons with the lattice structure of 2D atomic crystals, we characterized several 2D atomic crystals with oleamide assembly by combining STEM, SHG and polarized Raman spectroscopy. We first recorded the geometry of MoS$_2$ flakes with oleamide assembly by atomic force microscope (AFM) and then selectively transferred the same MoS$_2$ flake to a holey carbon grid for atomically resolved STEM imaging (Fig. 2a, b, see Supplementary Methods for experimental details). The correlation between the orientations of the nanoribbons and the MoS$_2$ atomic structures can be accurately affirmed by overlapping the geometric images taken by AFM and STEM (Supplementary Fig. 8a, b) and the results confirmed that oleamide nanoribbons oriented along the zigzag directions of MoS$_2$ lattice (see Supplementary Note 1 for more details). Besides STEM imaging, SHG was also utilized to characterize the lattice orientations of MoS$_2$ with oleamide nanoribbons. The angle-resolved SHG intensity measured on monolayer MoS$_2$ (Fig. 2c) with oleamide nanoribbons shows a clear six-petal pattern and the maximum petal direction points to the armchair lattice orientations of MoS$_2$ (Fig. 2d). Hence, the zigzag lattice orientations of the MoS$_2$ monolayer are determined by rotating the armchair directions by 30°, which are exactly the same as the orientations of the nanoribbons shown in Fig. 2c, again, confirming that oleamide nanoribbons align along the zigzag orientations of MoS$_2$. Besides MoS$_2$, we conducted SHG measurement on WSe$_2$ with oleamide nanoribbons and obtained the same conclusion (Supplementary Fig. 9).

Besides hexagonal 2D materials with threefold symmetry, we also explored the correlations between the nanoribbon orientations and the lattice structures of 2D black phosphorus
**Fig. 1** Self-assembly of oleamide on various 2D atomic crystals. 

**a** Schematic representation of oleamide self-assembly on 2D atomic crystals along specific orientations. For clarity, only the amide groups are displayed and the alkyl chains of oleamide are not shown except for the topmost oleamide dimer which shows the complete molecular structures. Hydrogen bonds are indicated with blue dashed lines. White, gray, blue and red balls represent H, C, N and O atoms, respectively.

**b-i** AFM images of oleamide nanoribbons on graphene, h-BN, MoS$_2$, MoSe$_2$, MoTe$_2$, WSe$_2$, black phosphorus (BP) and ReS$_2$, respectively. 

**j** Nanoribbons on CVD-grown multilayer MoS$_2$. Blue arrows represent the orientations of nanoribbons on varied layers. All scale bars are 500 nm, except for that in **c** which is 200 nm.
with tetragonal symmetry and ReS$_2$ with triclinic symmetry (Fig. 2e and Supplementary Fig. 10c) with atomically resolved STEM and angle-resolved polarized Raman spectroscopy. Following the similar STEM imaging process of MoS$_2$, the armchair orientation of black phosphorus is determined to be along the bisector of the nanoribbon directions, with rotations of $33^\circ$ to each nanoribbon orientations (Fig. 2e, f). Polarized Raman spectroscopy measurements on 2D black phosphorus with oleamide nanoribbons also revealed the same relationship between the orientations of nanoribbons and the armchair direction of black phosphorus (Fig. 2g, h)$^{27}$. Likewise, the nanoribbons assembled on ReS$_2$ were verified to be aligned along the direction of Re chains (zigzag lattice directions) using angle-resolved polarized Raman spectroscopy (Supplementary Fig. 10)$^{18}$. So far, we have demonstrated that the preferred orientations of oleamide nanoribbons can be well-correlated with the lattice structure of 2D atomic crystals experimentally.

### Theoretical calculations on the surface-templated assembly of oleamide

Next, we investigated the origins for the preferred orientations of oleamide nanoribbons on 2D atomic crystals from theoretical aspects. The orientation of an oleamide molecule on 2D atomic crystals is dictated by the van der Waals interactions between adsorbate and substrate. So we first calculated the rotation potential energy curve of a single oleamide molecule on monolayer MoS$_2$ with only Grimme’s D3 dispersion parameters (Supplementary Fig. 11) and the adsorption energy of a single oleamide molecule aligned along either zigzag or armchair direction of monolayer MoS$_2$ by the DFT-D3 method$^{28}$ (see Supplementary Methods for calculation details). For each alignment, we performed 2D potential energy surface (PES) scan to ascertain the most favorable adsorption position by accounting for only dispersion interactions between oleamide and MoS$_2$. The 2D PES for zigzag aligned oleamide and its 1D projection along the $x$ (zigzag) and $y$ (armchair) directions respectively have been shown in Fig. 3a, b. The energy valleys in Fig. 3a exhibit the same hexagonal symmetry as the underlying lattice of MoS$_2$. Our DFT-D3 calculations of the adsorption energy show that the zigzag aligned oleamide is favored by 0.11 eV as compared to the armchair aligned one. Because the interactions between oleamide molecules and the surface are stronger than the intermolecular interactions between oleamide molecules$^{26}$, the surface lattice will template the formation of nanostructures that cannot be formed in solution$^{25}$ (Supplementary Fig. 6). Thus the adsorbate-substrate interactions dictate the assembly of oleamide molecules on the surface of 2D atomic crystals and the tendency to achieve maximum H-bonding between –C=O and -NH$_2$ groups of adjacent molecules serves as secondary interactions$^{26}$. Based on the thermodynamically most stable adsorption position of individual zigzag aligned oleamide, we constructed the assembly pattern of oleamide molecules on MoS$_2$ (Fig. 3c). In this pattern, oleamide molecules not only form H-bonded dimers in a head-to-head configuration but also H-bonded nanoribbons in a side-by-side configuration. The orientations of nanoribbons are along the zigzag lattice directions of the underlying MoS$_2$, as also clearly seen from Fig. 3a. Our calculations also confirm that oleamide molecules form H-bonded nanoribbons on other hexagonal 2D atomic crystals including graphene, BN, MoSe$_2$, MoTe$_2$ and WS$_2$, along the zigzag lattice directions (Supplementary Fig. 12 and Supplementary Note 2). The first molecular layer of nanoribbons will in turn template the second layer assembly, and so on. The rotation and 2D PES scan showed that the second layer oleamide...
molecules and all layers above align along the same direction as the first molecular layer and stack right on top of the molecules in the bottom layers (Supplementary Fig. 13 and Supplementary Note 3). Similar calculations can be performed on other 2D atomic crystals to reveal their respective preferred adsorption directions.

Universal and nondestructive identification with oleamide. As both experimental and theoretical studies have confirmed that oleamide molecules can be used to probe the lattice orientations of various 2D atomic crystals, we further demonstrated the applications of this approach to identifying the edge orientations, grain domain and stacking rotations, which all significantly affect the intrinsic properties of these materials. Besides mechanically exfoliated 2D materials, well-aligned oleamide nanoribbons were also observed on chemical vapor deposition (CVD)-grown MoS2 regardless of the number of layers (Fig. 1j). With this approach, we identified that the edges of CVD-grown MoS2 flakes were along zigzag directions as the nanoribbons orientated parallel to the edges of the triangular CVD-grown MoS2 flakes, which is consistent with the STEM imaging (Fig. 1j and Supplementary Fig. 14). In addition to the as-grown edges, the MoS2 edges created by oxygen etching were also identified to be along zigzag directions of MoS2 lattice by using oleamide nanoribbons as probes (Supplementary Fig. 15a and Supplementary Note 4). Moreover, the grain domain in polycrystalline CVD-grown MoS2 and the interlayer rotation angles in multilayer MoS2 can also be easily identified with this approach (Supplementary Fig. 15b, c and Fig. 1j). Therefore, this approach is universal in determining the lattice orientations of various 2D atomic crystals, regardless of the chemical compositions, preparation methods and geometry. Besides being universal in identifying different kinds of 2D atomic crystals, the most prominent advantages of our identification approach is that it is simple and nondestructive. This identification process is very simple as only conventional AFM imaging is involved to observe the orientation of oleamide nanoribbons. This process can be further simplified to directly image the orientation of the oleamide assembly with an optical microscope as some of the nanoribbons can grow up to micrometer scale (Fig. 4b, c) as the result of molecule transfer from smaller nanoribbons with higher chemical potential to bigger ones with lower chemical potential, similar to the Ostwald ripening of crystals in a liquid phase (Fig. 4a)29, 30. More importantly, this approach is nondestructive as the melting point of oleamide is relatively low (~75 °C) and thus can desorb from the surface of the 2D atomic crystals during storage under ambient conditions (Supplementary Fig. 16). To speed up the desorption, we annealed the MoS2 samples with oleamide assembly in vacuum at 300 °C for 1 h and observed that all the nanoribbons vanished (Figs. 4d, e) without inducing obvious peak position shift in photoluminescence (PL) and Raman spectra (Fig. 4f, Supplementary Fig. 17 and Supplementary Note 5) and the surface roughness of the MoS2 flakes was identical to the pristine value after the removal (0.264 nm (before) and 0.269 nm (after)). To further rule out the possible degradation on the intrinsic properties of MoS2 by the identification process, we fabricated back-gated field effect transistors on both pristine and identified monolayer MoS2 (Supplementary Fig. 18) and compared their electrical performances. The averaged mobility
**Fig. 4** Optical visualization and nondestructive removal of oleamide nanoribbons. a, Schematic for the evolution of nanoribbons driven by chemical potential. Small nanoribbons shrink while the large ones grow up. b, c AFM images of oleamide nanoribbons on a few-layer MoS$_2$ flake after 1 h and 8 days under ambient condition, respectively. Scale bar, 1 µm. Inset Optical images. Scale bar, 2 µm. d, e AFM images of a monolayer MoS$_2$ flake with oleamide nanoribbons before and after annealing in vacuum at 300 °C for 1 h, respectively. Scale bars, 500 nm. f PL spectra collected from the same monolayer MoS$_2$: pristine (black), after oleamide self-assembly (red), and after the removal of nanoribbons (blue). g Typical $I_{ds}$-$V_{gs}$ curve for CVD-grown monolayer MoS$_2$ flakes after the removal of nanoribbons ($V_{ds}=1$ V). Inset The averaged mobility of pristine (blue) and identified MoS$_2$ after the removal of nanoribbons (red).
of monolayer CVD-grown MoS2 flakes that underwent oleamide assembly and removal is ~8 cm2 V−1 s−1, similar to the value obtained from their pristine counterparts (Fig. 4g), indicating that the intrinsic properties of underlying 2D atomic crystals can be well preserved throughout the whole processes.

**Discussion**

In summary, we explore the self-assembly of molecules on various 2D atomic crystals by using oleamide molecules as an example both from experimental and theoretical aspects. We show that the periodic atomic arrangements of these materials can guide the assembly of molecules into well-ordered supramolecular structures and correlate the orientations of the oleamide assembly with the lattice structure of the underlying 2D atomic crystals. With self-assembled oleamide nanoribbons as probes, the crystallographic orientations, grain boundaries and stacking configurations of a large variety of 2D atomic crystals regardless of preparation methods and geometry can be easily identified. More importantly, this approach is nondestructive as confirmed by both spectroscopic and electrical measurements, which enables further applications of the identified materials. By using molecules as probes, we open up a new way for simple and nondestructive identification of lattice structures of various substrates especially for 2D atomic crystals, which greatly facilitates both fundamental and application studies of these emerging materials.

**Methods**

**Preparation of 2D materials.** The 2D atomic crystals were obtained by mechanical exfoliation of bulk crystals purchased from 2D Semiconductors on SiO2/Si substrates with Scotch tape, followed by annealing in vacuum at 320 °C for 30 min to remove residual tape (290 °C for ReS2) before the assembly of oleamide. CVD-grown MoS2 flakes were obtained using electrochemical oxidized Mo foils as precursors and the growth was carried out at 650–800 °C.31

**Self-assembly of oleamide on 2D materials.** The assembly of oleamide was performed by spin coating 7 μL dilute solution (1.65 mmol L−1) of oleamide (Sigma-Aldrich, 99%) in chloroform (Alfa Aesar, 99%) on 2D materials at 2400 r.p.m.

**Characterization of nanoribbon/2D materials.** AFM images were captured with Bruker Dimension Icon in ScanAsyst mode. The optical images were taken with an Olympus BX 51 M microscope. Raman and PL spectra were collected with a Horiba-Jobin-Yvon Raman system under 532 nm laser excitation with a power of 2 mW and an Olympus BX 51 M microscope. Raman and PL spectra were collected with an Olympus BX 51 M microscope. Raman and PL spectra were collected with a Horiba-Jobin-Yvon Raman system under 532 nm laser excitation with a power of 2 mW. An electron microscope operated at 200 kV and equipped with double spherical aberration correction was used to study the formation of sheet-like nanoribbons. Angew Chem. Int. Ed. Engl. 54, 9857–9860 (2015).

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Author contributions
L.J. and J.W. conceived the ideas. L.J. and D.W. co-supervised this study. J.W., Z.L. and J.Z. performed the experiments. H.Y. and D.W. performed theoretical calculations. L.G. and X.L. performed STEM measurements and analysis. X.Z. and K.L. performed SHG measurements. R.Z. provided detailed instructions on the assembly of oleamide. L.J., J.W., D.W. and H.Y. co-wrote the manuscript. All the authors discussed the results and commented on the manuscript.

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