

Title	Strain-induced hierarchical ripples in MoS ₂ layers investigated by atomic force microscopy
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Publication date	2020-10-12
Original Citation	Hussain, S., Xu, R., Xu, K., Lei, L., Meng, L., Zheng, Z., Xing, S., Guo, J., Dong, H., Liaqat, A., Iqbal, M.A., Li, Y.J., Sugawara, Y., Pang, F., Ji, W., Xie, L. and Cheng, Z. (2020) 'Strain-induced hierarchical ripples in MoS ₂ layers investigated by atomic force microscopy', Applied Physics Letters, 117(15), p. 153102. Available at: https://doi.org/10.1063/5.0023405 .
Type of publication	Article (peer-reviewed)
Link to publisher's version	10.1063/5.0023405
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Download date	2026-10-29 01:32:29
Item downloaded from	https://hdl.handle.net/10468/15354



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Cite as: Appl. Phys. Lett. **117**, 153102 (2020); <https://doi.org/10.1063/5.0023405>

Submitted: 29 July 2020 . Accepted: 24 September 2020 . Published Online: 12 October 2020

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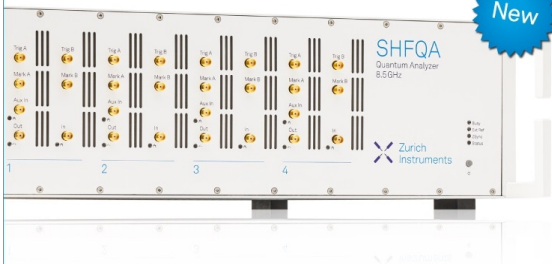
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Submitted: 29 July 2020 · Accepted: 24 September 2020 ·

Published Online: 12 October 2020



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ABSTRACT

Strain engineering plays a vital role in controlling the physical properties of two-dimensional (2D) materials. However, the nanomechanical behavior of atomically thin 2D crystals under strain has not been completely understood. Here, strain-induced hierarchical ripple nanostructures in triangular MoS₂ flakes were investigated by advanced atomic force microscopy and optical spectral measurements. The hierarchical nanoripples exhibited a threefold radial pattern, and their mechanical, electronic, and optical spectra characteristics were significantly modified due to the suffering from large tensile strain. Structure evolution of these hierarchical nanoripples was further discussed based on the geometry and thickness of MoS₂ flakes, and we attributed the curtain effect at the limit of a single atomic layer. Our study will be beneficial in designing nanomechanical structures and prototype electromechanical devices with 2D materials.

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Owing to their excellent electrical and mechanical properties, transition metal dichalcogenides (TMDCs) have emerged as an important member of two-dimensional (2D) layered materials.^{1–3} As a typical semiconducting TMDC, MoS₂ has garnered considerable research attention owing to its directive and appropriate bandgap, which makes it extremely useful for diverse applications in electronics and optoelectronics. Furthermore, its Young's modulus is ~270 GPa, and the breaking strength of defect-free monolayer MoS₂ can reach 11% of its Young's modulus.⁴ The exceptional mechanical properties of MoS₂ facilitate strain engineering as an effective method for controlling its electrical and optical properties. The applied strain can be used to modify the bandgap,^{5–7} conductivity, phonon modes,^{8–13} as well as photonic performances of these materials.

Diverse methods have been proposed for introducing strain in 2D materials, including bending of films on elastic substrates,^{7,15,16} stretching films by atomic force microscopy (AFM) probes,^{4,14,17,18} thermal expansion mismatch,^{19–21} and so on.^{22–25} Among the previous

strain engineering works, several groups focused on the strain-induced nanostructures in real space, such as ripples and wrinkles, and their specific physical properties.^{24,26–29}

As a convenient method, the thermal expansion coefficient (TEC) mismatch between the samples and substrate has been adopted for applying strain to TMDC specimens through the fast cooling process after the CVD growth.³⁰ Meanwhile, the TMDC flakes with varying geometries and shapes can be controllably synthesized.^{31–33} In this study, we focused on the strain-induced local nanostructures of the CVD-grown triangular MoS₂ flakes through the above TEC mismatch method. The threefold hierarchical ripple nanostructures were clearly imaged and extensively investigated by a combination of functional AFM, Raman, and photoluminescence (PL) spectra measurements. The specific mechanical and electrical properties of these specific ripple structures were experimentally detected, which are due to the larger tensile strain in these regions. Their structure and property evolutions were further discussed based on the geometry and thickness

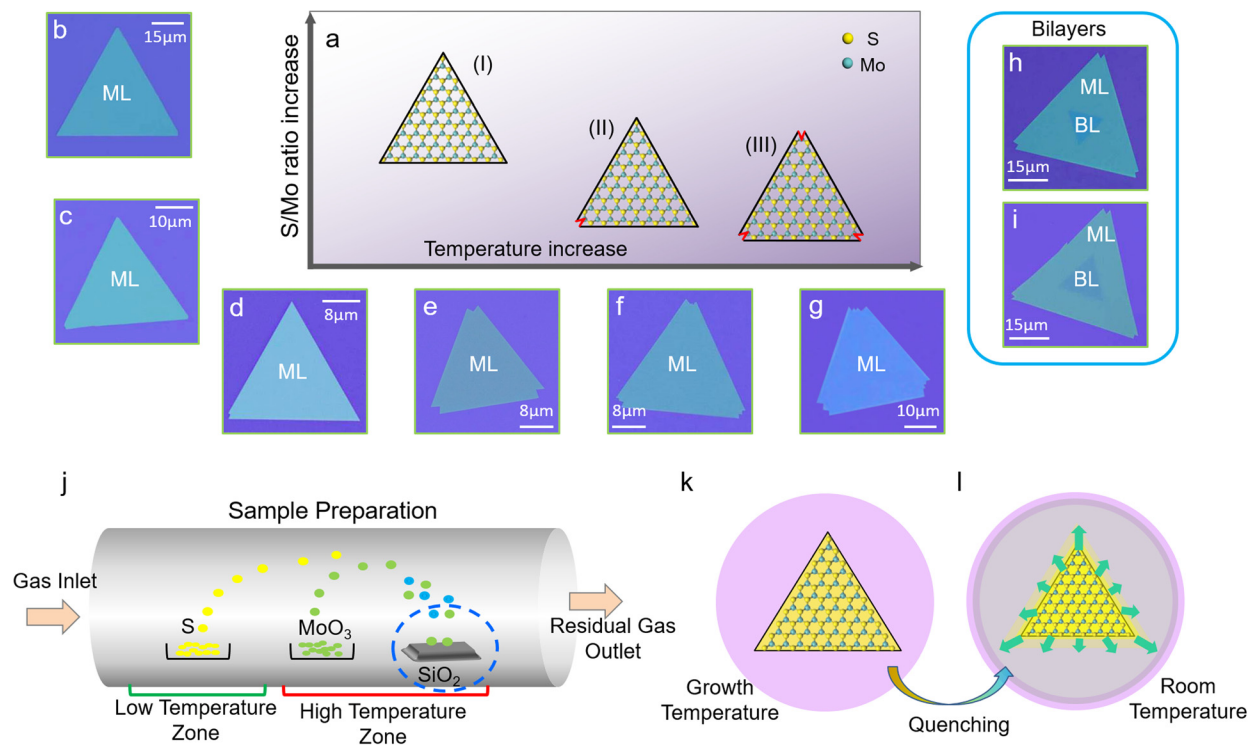


FIG. 1. Growth of triangular MoS₂ flakes and the introduction of tensile strain. (a) Schematic for the shape evolution of MoS₂ flakes with various S/Mo ratios and growth temperatures. (b)–(h) Optical topographic images of a series of typical triangular flakes with multi-apex corners. (j) Schematic of the CVD growth process. (k)–(l) The introduction of tensile strain applied by the substrate during the fast cooling process.

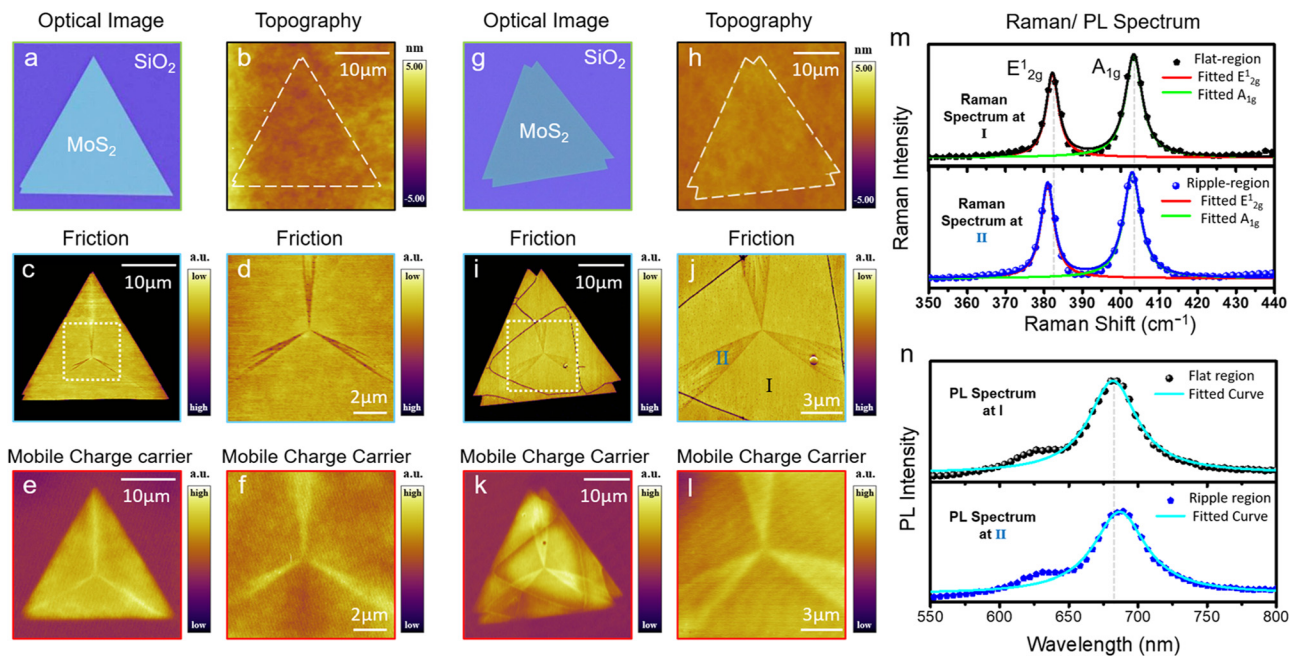


FIG. 2. Topography and physical properties of thermal-strain-engineered triangular MoS₂ flakes. (a) and (b) Topographic, (c) and (d) friction, and (e) and (f) mobile charge carrier images of the MoS₂ flake with small dual-apex corners. (g) and (h) Topographic, (i) and (j) frictional, and (k) and (l) mobile charge carrier images of the MoS₂ flake with larger dual-apex corners. (m) Raman and (n) PL spectra for the flat and ripple regions, which are marked with I and II in (j).

of MoS₂ flakes. We attributed these hierarchical ripple nanostructures to the modified curtain effect of the triangular MoS₂ flakes.

The shape of the MoS₂ flakes can be modified by controlling the S/Mo ratio and growth temperature, as shown in Fig. 1(a). The normal triangular flakes are the most common ones, which are obtained when the S/Mo ratio is high and growth temperature is relatively low. When the S/Mo ratio is reduced and growth temperature is increased, triangular flakes with multi-apex corners are obtained, as shown in Figs. 1(b)–1(g). The optical image of each flake shows a uniform contrast. Furthermore, the second layer can grow at the center of these triangular flakes and exhibits a perfect triangular shape. Both 2H- and 3R-MoS₂ bilayer polytypes were obtained, as shown in Figs. 1(h) and 1(i). After the CVD growth process at high temperature, the isotropic tensile strain was applied on the triangular MoS₂ flakes through the substrate during the fast cooling process. The tensile strain is due to the TEC mismatch between MoS₂ flakes (10^{-5} K^{-1}) and the SiO₂/Si substrate (10^{-7} K^{-1})²⁹ and may induce the out-of-plane ripples due to

the Poisson instability³⁴ of MoS₂ layers. Considering the geometry of MoS₂ flakes, the strain at the three corners was larger than in other regions, as shown in Fig. 1(l).

Figure 2 shows two typical triangular MoS₂ monolayer flakes with one small dual-apex corner [Fig. 2(a)] and three large dual-apex corners [Fig. 2(g)]. The optical images and AFM topographies reveal a uniform contrast of MoS₂ monolayers. In our previous work,³⁵ the transverse shear microscopy (TSM) mode has been developed as a convenient method to image the domain structures of polycrystalline MoS₂ flakes (Fig. S1). Here, all of these triangular flakes with multi-apex corners have been determined as single crystalline by the TSM measurements (Fig. S2). The distinctive strain-induced features were clearly observed by the friction force microscopy (FFM) mode,²⁶ as shown in Figs. 2(c) and 2(i), exhibiting a threefold radial pattern from the center of triangular MoS₂ monolayer flakes.^{36,37} The symmetry of the features was affected by the strain distribution within the flakes, which depended on the geometry of MoS₂ layers [Fig. 1(l)]. In the

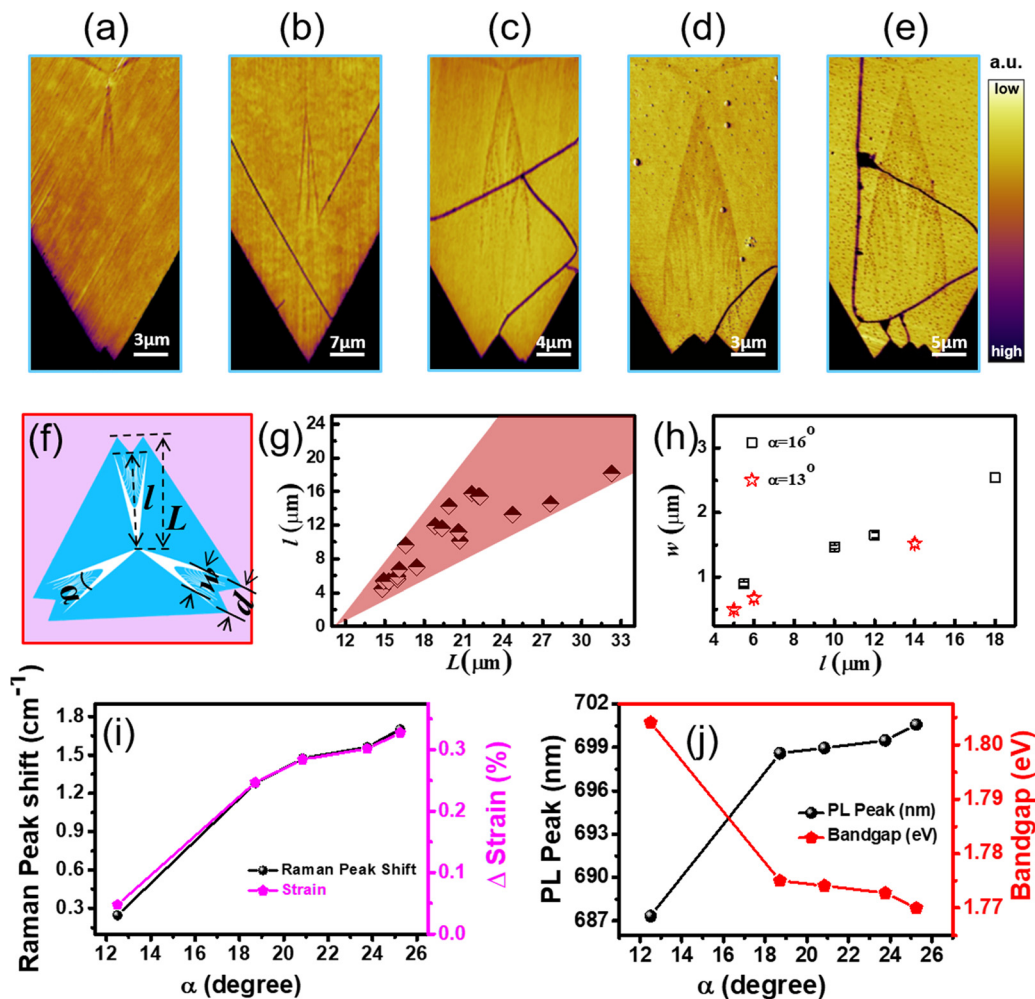


FIG. 3. The dependence and evolution of hierarchical ripple structures on the geometry of MoS₂ flakes. (a)–(e) FFM images of a series of hierarchical ripple structures. (f) Schematic definitions of the opening angle α , the length l , and width w of the hierarchical ripple structure, the width d of multi-apex corner, and the size L of triangular flakes. (g) Statistics of l vs L . (h) Statistics of w vs l . (i) Raman shift of E_{2g}^1 mode and strain difference vs α . (j) PL peak wavelength and bandgap vs α .

close-up FFM images [Figs. 2(d) and 2(j)], the hierarchical ripple nanostructures were clearly shown in these strain-induced features [marked with II in Fig. 2(j)], named ripple regions, distinctively different from the flat regions [marked with I in Fig. 2(j)] (see more hierarchical ripple nanostructures in Fig. S7). The indiscernible nanoripples in AFM topographies imply that the height of the ripples was less than 1 nm.²⁶ It is also noted that these nanoripple areas exhibited lower friction signals, which should be due to the strain-modified mechanical properties of MoS₂ films.

According to the previous theoretical works, the tensile (or compressive) strain can decrease (or increase) their Young's modulus. It can be speculated that these hierarchical ripple structures will show lower elastic modulus than the flat areas within the MoS₂ flakes. Bimodal AM-AM AFM mode³⁸ was further employed to evaluate the elastic modulus (Figs. S3 and S4). First, the Young's moduli of the SiO₂/Si substrate and unstrained MoS₂ are determined as ~ 75 GPa and ~ 268 GPa, which is consistent with a previous study.³⁹ Then, the Young's modulus of hierarchical ripple areas is determined as ~ 222 GPa, which is only $\sim 83\%$ of that of flat MoS₂ regions and consistent with our speculations.

In addition to their local mechanical properties, the local electronic properties can also be modified by the in-plane strain within these MoS₂ films. It has been reported that tensile (or compressive)

strain can decrease (or increase) the bandgap of MoS₂ films.⁴⁰ The DH-EFM⁴¹ mode (Fig. S5) was further employed to qualitatively evaluate the local mobile charge carrier density and bandgaps of MoS₂ films.^{41,42} As shown in Figs. 2(e), 2(f), and 2(k) and 2(l), the higher mobile charge carrier densities are clearly observed at the hierarchical ripple areas than the flat regions within the triangular flakes, indicating the decreased bandgap of these ripple structures under the tensile strain.

Probing the shift in phonon frequencies is also an effective way to investigate the strain effect on MoS₂ films. Raman spectra for the flat and hierarchical ripple regions of MoS₂ flakes are shown in Fig. 2(m). It is known that A_{1g} and E_{2g} modes are associated with the out-of-plane and in-plane vibrations of sulfur atoms, respectively. Here, the A_{1g} mode is less affected in terms of softening and broadening of the peak than the E_{2g} mode.⁴³ In contrast to the unstrained MoS₂ layers (385 cm⁻¹),⁴⁴ the E_{2g} mode of flat and ripple regions is shifted toward lower frequencies of 380.9 cm⁻¹ and 382.1 cm⁻¹, respectively. It is clearly indicated that both flat and ripple regions are under the tensile strain, and the ripple region suffers much larger tensile strain than the flat regions.

Furthermore, the effect of tensile strain on the band structure of MoS₂ monolayers was studied by PL spectroscopy. It is well known that the intrinsic MoS₂ monolayer is a direct bandgap semiconductor

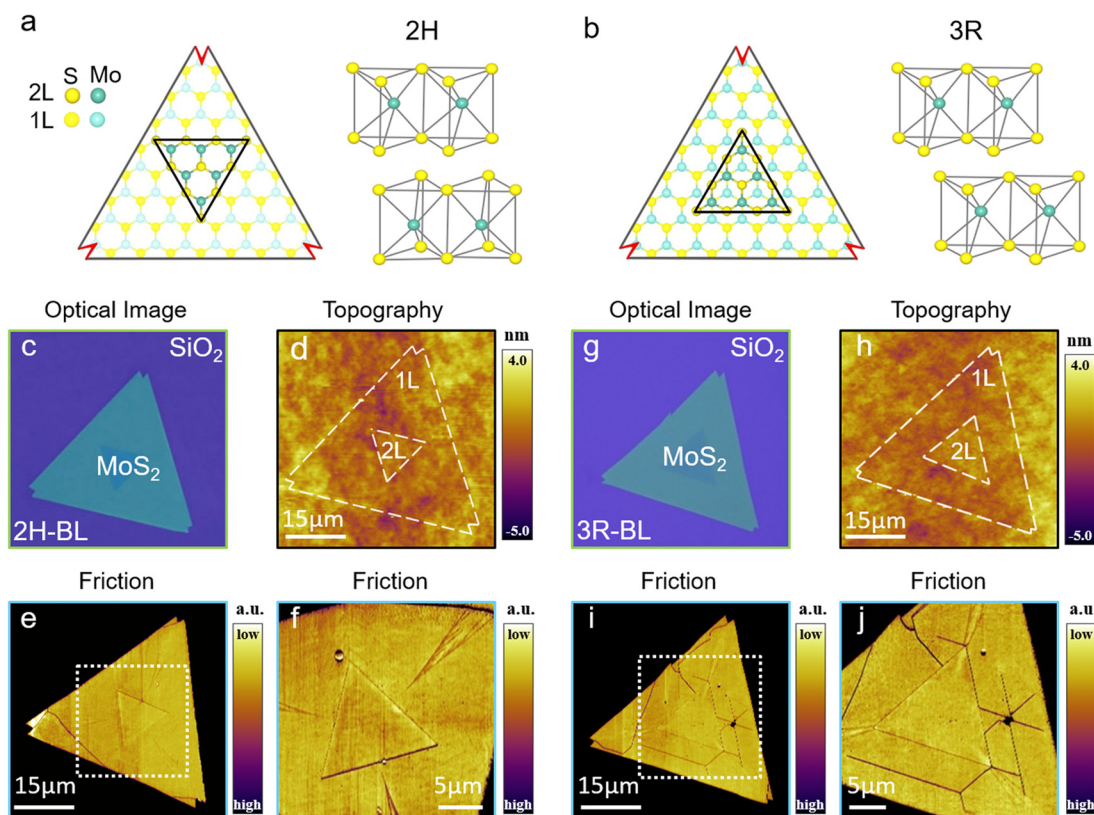


FIG. 4. Strain-induced hierarchical ripple structures of the MoS₂ bilayer flakes. (a) and (b) The crystalline structures of (a) 2H- and (b) 3R-MoS₂ bilayer polytypes. (c)–(f) Optical (c), topographic (d), and FFM (e) and (f) images of the 2H-MoS₂ bilayer flake. (g)–(j) Optical (g), topographic (h), and FFM (i) and (j) images of the 3R-MoS₂ bilayer flake.

with a bandgap of ~ 1.82 eV. When a tensile strain is applied on the MoS₂ monolayer, the PL peak exhibits a redshift (i.e., smaller bandgap) with respect to the unstrained MoS₂ monolayer. Based on the corresponding PL peaks, the bandgaps of ripple and flat regions were determined as ~ 1.775 eV and ~ 1.815 eV, respectively. The PL results also confirm that the ripple region suffers larger tensile strain than the flat parts of MoS₂, which is consistent with all of the above DH-EFM analysis.

All of the above results indicate that the hierarchical ripple regions suffer larger tensile strain than the flat regions although the substrate applies a uniform tensile stress on the MoS₂ flakes. According to the extensive FFM measurements of various triangular flakes (Fig. S7), it can be supposed that these hierarchical ripple nanostructures should be related to the geometric shape of the MoS₂ flakes, especially their multi-apex corners. Then, we can speculate that the specific ripple nanostructures could be obtained by controlling the MoS₂ flakes. As a first step, we should discuss the dependence and evolution of hierarchical ripple nanostructures on the size and geometric shape of the MoS₂ flakes.

Figures 3(a)–3(e) show a small part of the FFM images for the hierarchical ripple structures with varying multi-apex corners. In Fig. 3(f), the geometric parameters of the hierarchical ripple structure and triangular MoS₂ flake are schematically defined. According to the statistics, the size L of triangle flakes is a crucial parameter for the formation of ripple structures, as shown in Fig. 3(g). The ripple structures can only form when L is larger than ~ 11 μm . The length l of ripple structures increases monotonically with the size L of triangle flakes. At the same opening angle α , the corresponding width w is linearly proportional to the length l of ripple structures, as shown in Fig. 3(h). Raman and PL spectra were further employed to investigate these typical ripple structures (Fig. S8). It is evident that the strain difference (Δ_{strain}) between the flat and ripple regions increases as the opening angle α increases, as shown in Fig. 3(i). Furthermore, we also observed that the bandgap of the ripple structures reduces as the opening angle α increases, as shown in Fig. 3(j). The above experimental results imply that the size of hierarchical ripple structures is roughly determined by the size of triangular flakes, while their strain-induced properties are mostly controlled by the opening angle α or the width d of multi-apex corners of the flakes.

Except for the size and shape of the triangular flake, the thickness of MoS₂ also can affect the hierarchical ripple structures. MoS₂ has three common polytypes, which vary by stacking order and atom coordination. The 2H and 3R structures occur in nature and have trigonal prismatic coordination.² The crystalline structures of 2H- and 3R-MoS₂ bilayer polytypes are shown in Figs. 4(a) and 4(b), respectively. No distinctive features but uniform contrast was observed within these bilayer flakes in the optical and AFM topographic images. However, the threefold strain-induced ripple patterns in both 2H- and 3R-MoS₂ bilayers were observed, as shown in the FFM images of Figs. 3(e) and 3(i).

Figures 4(f) and 4(j) show the magnified images of the hierarchical ripple structures. It is noted that the nanoripples are located only at the first layer and shifted away from the central bilayer areas of the flakes. This phenomenon could be attributed to two reasons. First, Young's modulus of the bilayer is larger than that of the monolayer ($E_{2L} > E_{1L}$).¹⁸ Hence, the central bilayer regions of Figs. 4(e) and 4(i) are hard to deform and immune to nanoripples.

Second, the bottom layers are directly subjected to the substrate tensile strain, which is applied during the fast cooling process. Therefore, the top layers are only subjected to much smaller tensile strain through the weak in-plane interlayer interactions. The smaller bandgap and higher carrier densities at these tensile strain-induced ripple areas were also confirmed by the DH-EFM measurements (Fig. S9).

It is well known that the universal self-similar hierarchy of ripples can be observed within the thin sheets or films when they are under tensile strain and boundary edge confinement.^{34,45–50} The so-called curtain effect is clearly demonstrated in our simple experiment of Fig. 5(d). These ripples are mainly along the longitudinal direction (the same as the direction of gravity) but perpendicular to the boundary edges. Wrinklon is the localized transition zone where two ripples merge, which is a building block for these hierarchical ripple patterns. To further understand their structural evolution, we gradually bent the boundary edge of this curtain, as shown in Figs. 5(a)–5(c). When the edges are bent to form a “ $\wedge$ ” shape, all the ripples were not strictly orienting along the longitudinal direction any more but affected by the bent edges. The ripples near the edges still try to orient along the perpendicular direction of the bent edges. When the ripples are far away from the edges, they gradually bend along the longitudinal tensile direction of gravity and still retain hierarchy. It is noted that the hierarchical ripples in the bent curtains are relatively similar to those in the

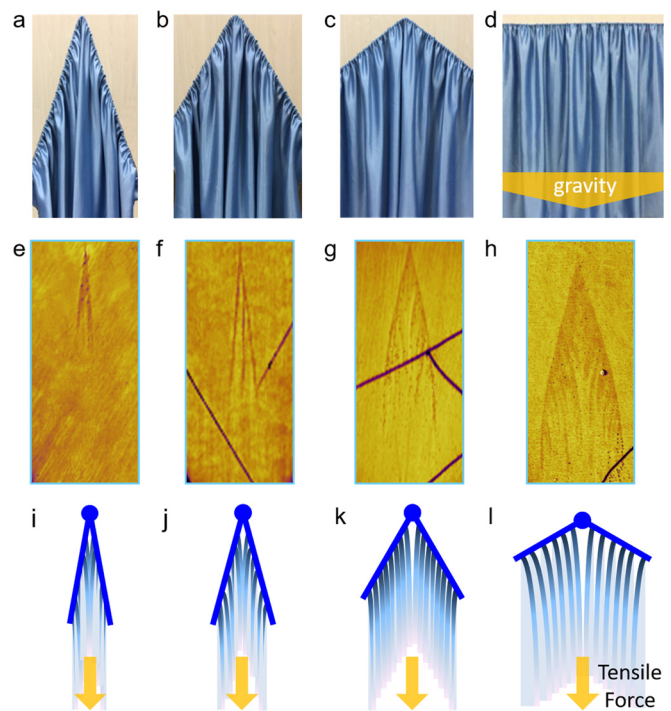


FIG. 5. The formation mechanism of hierarchical ripple structures. (a)–(d) The evolution of hierarchical ripples in a silk curtain when the confined edges are gradually bent. These ripples are generated through the longitudinal tensile strains applied by the gravity of itself. (e)–(h) FFM images of the hierarchical ripple structures in MoS₂ with different opening angles. (i)–(l) The schematic model for elucidating the formation and evolution of hierarchical ripples.

triangular MoS₂ flakes. Based on their inherent similarities, a simple diagram is proposed to elucidate their formation mechanism, as shown in Figs. 5(i)–5(l). More theoretical work will be needed to understand the formation mechanism of these complicated hierarchical ripples, which is out of the scope of this paper at here.

In summary, the thermal-strain-induced hierarchical ripple structures within the triangular MoS₂ flakes have been extensively investigated by a combination of functional AFM, Raman, and PL spectra measurements. The lower Young's modulus and smaller bandgaps of these ripple structures were experimentally determined, which are due to the suffering of larger tensile strain in these regions. The evolution and dependence of these hierarchical ripple structures were further discussed on the geometry and thickness of MoS₂ flakes. Finally, the modified curtain effect has been proposed to understand the formation of hierarchical ripples. Our study will be beneficial in designing nanomechanical structures and prototype electromechanical devices based on emerging functional two-dimensional materials.

See the [supplementary material](#) for additional details on the AFM characterization techniques, material preparation method, Raman/PL Spectrum, and some [supplementary material](#).

This work was supported by the National Natural Science Foundation of China (NSFC; Grant Nos. 21622304, 61674045, 11604063, 61911540074, 11622437, and 11974422); Ministry of Science and Technology (MOST) of China (Grant Nos. 2016YFA0200700 and 2018YFE0202700); Strategic Priority Research Program, Key Research Program of Frontier Sciences and Instrument Developing Project of Chinese Academy of Sciences (CAS; Grant Nos. XDB30000000, QYZDB-SSW-SYS031, and YZ201418); Grant-in-Aid for Scientific Research from Japan Society for the Promotion of Science (JSPS) from the Ministry of Education, Culture, Sports, Science, and Technology of Japan (Nos. JP16H06327, JP16H06504, JP17H01061, and JP17H010610); and Osaka University's International Joint Research Promotion Program (Nos. J171013014, J171013007, J181013006, and Ja19990011). Z. H. Cheng was supported by Distinguished Technical Talents Project and Youth Innovation Promotion Association CAS, the Fundamental Research Funds for the Central Universities, and the Research Funds of Renmin University of China (Grant No. 18XNLG01).

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its [supplementary material](#).

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