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Striped Nanoscale Friction and Edge Rigidity of MoS₂ Layers

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Lateral force microscopy (LFM) is used to probe the nanoscale elastic and frictional characteristics of molybdenum disulfide (MoS₂). We find that MoS₂ edges are effectively flexed over a region of about 10 nm when scanned with sharp single asperity LFM probes, with energies consistent with out-of-plane bending and being slightly stiffer than those of graphene. Additionally, we report the first observation of a striped nanoscale frictional phase on the surface of MoS₂. This frictional phase is fixed to the underlying MoS₂ with a modulation length of ~4 nm that is insensitive to scan parameters and has domain sizes that exceed 100 nm. The amplitude of these features is found to be relatively independent of the geometry of the tip asperity and the applied load within the ranges we investigate. Experimental results suggest this periodic friction can be explained by variations in the local strain in the underlying MoS₂. These results could have general applicability to understanding the nanomechanical properties of the growing array of laminar materials that are of potential use as atomically-thin coatings to future nanoscale machines.

Introduction

There is tremendous interest in reducing the overall size of machines and their components to attain the smallest moving devices possible.[1] However, at these extremely reduced dimensions the mechanical properties of the nanoscale surfaces brings new challenges in their control and understanding.[2, 3] As machines are reduced in size towards this nanoscale regime, atomically-thin laminar materials are becoming increasingly attractive as ultra-thin coatings that could modify and protect the mechanical component surface properties (such as the local friction and elasticity), while maintaining the overall nanoscale topographical features.[4-8] Two of the most important laminar materials in modifying surface mechanical properties are graphite and MoS₂, as these have long been used as dry solid lubricants in large-scale applications due to the ability of their van der Waals (vdW) coupled layers to slide against each other.[9] There has long been interest in the behavior of few-layer and atomically-thin films of such laminar materials[10, 11] which has received recent extensive renewed interest.[12] Moreover, using these materials as atomically-thin mechanical coatings has been made much more achievable due to recent advances in the synthesis[13] and controlled placement[14] of few-layer films of these laminar materials. To fully exploit this growing class of potential atomicallythin nanomechanical coatings, it is critically important that the nanoscale mechanical properties of these materials be understood.[5]

Towards this effort, there have been a number of advances in understanding the nanoscale mechanical properties of these atomically-thin laminar materials. This includes single asperity investigations of laminar materials' frictional properties, [15, 16] extreme strength and elastic properties, [17] delamination, [18, 19] anisotropic friction and puckering of their surfaces, [16, 20] and localized frictional spikes at their step edges.[21, 22] It has also been demonstrated that multi-layer[23] and single layer[21] films of these laminar materials can effectively eliminate underlying nanoscale frictional spikes associated with step edges when they are used as coatings. Recent work has shown that covered edges of these materials have very different physical and chemical properties in comparison to exposed ones[24, 25] which show increased friction in the presence of absorbed water.[26] Moreover, the sharpness of the asperity in contact with an edge has been shown to play an important role in the local mechanical properties in the vicinity of a laminar material's edge.[21, 25, 27, 28] Through the use of an extremely sharp asperity it was shown that an exposed edge of graphene could be flexed and result in its stickslip,[21] as also suggested by molecular dynamic simulations.[29] Possibly related to this behavior at step edges have been recent reports of a striped stick-slip phase on graphite surfaces that is thought to result from the localized puckering of its surface.[30, 31] While anisotropic sliding has been observed on MoS₂[32] (as it has on graphite surfaces[33]), localized nanoscale stick-slip and periodic frictional modulations have not yet been observed in this laminar material.

Here we report the elasticity of MOS_2 edges and a striped frictional phase at the nanometer scale in this laminar material. We find that sharp lateral force microscopy (LFM) tips can effectively flex the edges of MOS_2 , with results consistent with out-of-plane bending. These results show that MOS_2 edges are slightly stiffer compared to those of graphene, a result which may be partly due to

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the stronger vdW interactions of MoS_2 layers. We also observe a periodic striped frictional phase on MoS_2 surfaces having a wavelength of ~4 nm. The striped frictional phase is shown to be robust over regions greater than ~100 nm and remain fixed to the underlying MoS_2 upon variation of the fast scan direction. Our experimental results suggest that this nanoscale periodic frictional phase of MoS_2 could be due to a striped strain field that varies the local puckering of the atomically-thin material in the presence of an asperity, and as a result modifies the local friction. Such robust modulations of an atomically-thin material could have application in controlling the surface mechanical properties at the nanoscale with long-range order.

Experimental details

Sample preparation

Silicon substrates with a 300 nm oxide layer were placed in an ultrasonic cleaner with acetone, isopropyl alcohol, and deionized water for 3 minutes each, followed by UV-ozone cleaning in a NovaScan PSD Series Digital UV Ozone System for 15 minutes. MoS_2 (obtained through SPI Supplies) was then mechanically exfoliated onto the silicon substrates. After exfoliation, the samples underwent CVD processing consisting of a constant gas flow rate of 380 sccm Ar and 340 sccm H₂ at 400 °C for one hour to remove tape residue.

Lateral force microscopy measurements

The LFM measurements were performed using an Asylum Research MFP-3D atomic force microscope (AFM) with PPP-LFMR and PPP-CONTSCR probes manufactured by Nanosensors. Both types of probes have nominal force constants of 0.2 N/m, resonant frequencies of 23 kHz, and a tip radius of curvature less than 10 nm. During LFM operation the probe is placed in contact with the sample surface and the fast scan direction is chosen to be perpendicular to the cantilever beam while both lateral and vertical deflections are measured. Prior to each LFM measurement the adhesion force between the probe tip and the sample is measured. During each scan the net force, F_{net} is kept constant. Here, F_{net} is defined as the sum of the normal load applied to the tip, L, and the adhesion force between the tip and the surface. All measurements were taken in ambient laboratory conditions (temperature ~ 20 °C).

To investigate the frictional response of MoS₂ edges we start with sharp LFM probes, as determined through measurement of the probes' adhesion force,[21] and focus on single layer MoS₂ step edges. LFM probes are observed to catch atomic step edges when the adhesion force is ≤ 2 nN. In order to maintain the sharp probes, we limit LFM scan sizes to < 1 μ m, and avoid scanning over the SiO₂ substrate, which is observed to increase the adhesion force. LFM scans are performed with the fast scan direction perpendicular to the cantilever. Both the lateral and vertical deflections of the probe are monitored with a laser that reflects off the cantilever and onto a four-quadrant position sensitive detector (PSD). The vertical

deflection is controlled through a feedback loop in order to maintain a constant load force as well as provide topographical information. As an LFM probe is scanned along a surface, lateral forces acting on the probe cause torsional rotation of the cantilever, resulting in a lateral deflection of the laser spot at the PSD.

Calibration of the lateral voltage signal (V_{lat}) is performed using a low load method introduced in Ref. [21] that assumes a vertical adhesion for atomically-thin laminar materials, rather than the usual normal adhesion.[34, 35] Since this calibration technique is performed at low loads, ~4 nN, and over atomically smooth materials, the probes remain sharp and exhibit minimal changes in adhesion over the course of the calibration.

Results and discussion

Elastic response of MoS₂ edges

Figure 1 shows the results of lateral force microscopy (LFM) measurements of a MoS₂ step edge consisting of 10 layers on the left of a step (measured to be ~6.5 nm thick relative to the SiO₂ substrate using an AFM) and 9 layers on the right (measured to be ~5.9 nm thick), as schematically illustrated in Fig. 1(a). Figure 1(b) shows a corresponding round-trip line scan of the measurement across the step-edge with ${\it F}_{net}=8.26$ nN, where the blue line is the "trace" scan from left to right, while the red "retrace" scan is from right to left. For this low-adhesion tip (with A = 1.8 nN) we observe LFM measurements that demonstrate stick-slip behavior for F_{net} loads greater than ~4 nN. The line scan in Fig. 1(b) shows an ~11 nm wide region of increased signal at the MoS₂ edge in the retrace (stepping-up) direction accompanied with a large lateral signal, while a significantly smaller signal is observed in the trace (stepping-down) direction. Moreover, the larger retrace signal has a roughly linear signal as a function of distance, as expected for a spring obeying Hooke's law. Such behavior is consistently observed at the MoS₂ edge, as seen in the LFM trace and retrace scan images in Figs. 1(c) and 1(d), respectively. The graded dark region in Fig. 1(d) is the spring loading ("stick") of the MoS₂ edge while the abrupt contrast change on its left is the MoS₂ edge abruptly "slipping" under the LFM tip.

Using the calibration of the LFM tip, as discussed above in *Experimental details*, we are able to deduce the effective spring constant through the slope of the LFM retrace signals, as in Fig. 1(b), which gives $k = 0.350 \pm 0.063$ nN/nm for the MoS₂ edge. This spring constant and the displacement yield a stored elastic energy of ½ $k x^2 \approx 132$ eV, which is similar to the values seen for graphene (~90 eV).[21] Given reported values of the Young's modulus $E \approx 0.27$ TPa, Poisson ratio $v \approx 0.27$, and thickness t = 0.65 nm of MoS₂, we estimate the in-plane strain energy for an $x \approx 10$ nm displacement as $\frac{\pi t}{8} \frac{E}{1-v^2} x^2 \approx 46,000$ eV.[21, 36] This energy is much larger than that measured with the cantilever, indicating the MoS₂ edge is likely bending out-of-plane (as depicted in Fig. 1(a)), rather

than in-plane.[21] The fact that the effective spring constant for flexing the MoS_2 edge is slightly larger than that measured for graphene (0.29 ± 0.11 N/m [8]) may be partially due to the larger vdW adhesion of MoS_2 (2.1 eV/nm²) as compared to graphene (0.9 eV/nm²).[37]

Insight into the stick-slip response of the MoS₂ step edge can be obtained by using the model for bending laminar materials proposed in Ref. [21] which is based on the approximate low-load analytic form of the Schwoebel-Ehrlich energy barrier in the vicinity of an atomic step-edge.[28, 38, 39] This energy barrier is approximated by $U = \mathcal{E}\left\{-erf(x/b_1) + erf([x-c]/b_2)\right\}, [28]$ where ${\cal E}$ is a constant of order an eV, b_1 represents the effective width of the MoS₂ edge ($x \equiv 0$), which should be on the order of the tip apex radius, and b_2 and c are constants which represent a slow recovery of the potential away from the edge which should be larger than b_1 . For a tip moving into a step edge, the slip point occurs at the point where $\frac{d^2U}{dx^2} = -k$. Previously, it was found that assuming an atomically-sharp asperity of $b_1 pprox 0.1$ nm (with $b_2 = c = 10b_1$) and a reasonable energy barrier of $\mathcal{E} = 2$ eV for an atomically-thin segment of a graphene edge yields a stick-slip distance of ~11 nm, in good agreement with the experimentally observed value.[21] For the case of a MoS₂ edge using the same normal load, the calculation of the stick-slip distance must be modified by using the slightly larger measured spring constant. In addition, one could expect a larger energy barrier at the MoS₂ edge in comparison to a graphene edge, since the physical step height is greater. To account for this larger barrier we take \mathcal{E} of the MoS₂ edge to be the value used previously for graphene scaled by the ratio of the thicknesses, i.e., $\mathcal{E} = \frac{0.65 \, nm}{0.335 \, nm} \times 2.0 \, \text{eV} \approx 3.9 \, \text{eV}$. This modified barrier height, combined with the measured spring constant yield an expected stick-slip distance of ~19 nm, in order of magnitude agreement with the ~11 nm observed experimentally in Fig. 1. The deviation between the calculated and measured stickslip distances is likely due to the rough estimate of the barrier energy, \mathcal{E} , and width, b_1 .

Nanoscale frictional stripes

In the vicinity of the MoS₂ edges we also observe modulated nanoscale frictional stripes. Such stripes are slightly apparent in the LFM trace image of Fig.1(c), but become easier to discern as F_{net} is decreased. Figure 2 shows LFM images obtained with a $F_{net} = 1$ nN where the frictional stripes are clearly discernable for both the trace (Fig. 2(a)) and retrace (Fig. 2(b)) scans. These features consist of modulations of the lateral signal with a wavelength of ~4.1 nm, an order of magnitude larger than the MoS₂ lattice constant (0.315 nm). Figures 2(c) and (d) show the lateral signal at the locations marked by the blue and red lines in Figs. 2(a) and (b). Although crossing the step edge results in an abrupt change in angle of the frictional stripes by approximately 57°, it does not result in a noticeable change in the modulation length scale (i.e., the wavelength). Moreover, these frictional features appear continuous over the scan region and show long-range order greater than ~100 nm. It is likely that the relative orientation of the stripes is related to the hexagonal symmetry of the MoS_2 . While the observed angles are not exactly at 60 degree intervals, this slight orientational deviation from that expected for the six-fold symmetric MoS_2 lattice is consistent with the reported angular deviations of stripes observed on graphene, which are thought to arise from slight grain boundary misalignments. [30]

This striped frictional phase appears robust to various methods of probing its LFM response. For example, the scan rate of the LFM measurement over the MoS₂ surface appears to make no noticeable effect, as seen in Fig. 3. Despite a factor of three variation in scan rate (from 0.15 Hz to 0.45 Hz) in going from Fig. 3(a) to 3(c), there is no appreciably change in the wavelength of these features, as verified through the use of a 2D Fast Fourier Transform (2D FFT) in Fig. 3(d). This FFT shows peaks at wave number magnitudes that are all approximately $0.25 nm^{-1}$, thus having the same wavelength regardless of scan rate, as shown in Fig. 3(e). The identification of the peaks in the FFT with the stripes in the LFM images is confirmed by selectively eliminating these Fourier components (circled in the FFT inset in Fig. 3(f)) and then taking the inverse transform, which (as a result) no longer contains the associated stripes on the right-hand side of the step. The fact that the wavelength is independent of scan rate indicates that these nanoscale frictional stripes are not due to extrinsic artifacts, such as periodic noise in the imaging system. More evidence that the stripes do not depend strongly on the details of the probe comes from the fact that LFM tips with different adhesions measure the same (~4.1 nm) periodicity, as seen in Fig. 4.

The amplitude of these frictional stripes also appears relatively insensitive to the scan parameters. In Fig. 5(a) we plot the RMS modulation of the lateral signal associated with the stripes (δV_{lat}) and the calibrated lateral force (δF_{lat}) as a function of scan rate in the vicinity of the step edge. (δV_{lat} is determined by taking the square root of the integral of the modulus squared of the associated FFT peaks.) This plot shows that the amplitude of the stripes is relatively insensitive to the speed of the LFM tip. Likewise, Fig. 5(b) also shows that the overall net load of the tip, F_{net} (from 1.42 nN to 8.26 nN), does not have an appreciable effect on the amplitude of the stripes. In contrast, the background average frictional force between the surface and the probe increases roughly linearly, with the form $F_f \approx \mu F_{net}$, where μ is found to be ~ 0.022. Thus, as shown in Fig. 5(c), the relative size of the modulated lateral force due to the stripes to the background frictional signal, $\delta F_{lat}/F_f$, decreases in magnitude as F_{net} is increased. This variation in the ratio is likely the reason that the striped frictional phase is much more readily apparent at low F_{net} .

Also attesting to the intrinsic nature of the lateral force modulations is the fact that the stripes appear fixed to the MoS_2 lattice, as is shown by rotating the sample below the tip. Figure 6(a) shows a LFM retrace image with the fast scan direction perpendicular to the step edge. The frictional variation on either side of the step edge are approximately \pm 30° from the vertical. Upon rotation of the sample by ~ 30° clockwise (Fig. 6(b)) the features to the right of the step edge are perpendicular to the fast scan direction. Thus, they appear to rotate with the sample and

their relative orientation is determined by the MoS_2 surface, rather than the scan angle of the tip (which remains the same between Figs. 6(a) and 6(b)). It is also apparent from the rotated scan in Fig. 6(b) that the amplitude of the stripes is dependent on their orientation with respect to the scan direction. This nanoscale frictional dependence on the scan angle could be related to the long-range (average) frictional dependence of single asperity tips on a strained and puckered surface of graphene, which has also been shown to be anisotropic.[20]

Over the range of several hundred nanometers these modulated frictional features we observe appear to be ordered with some domains greater than 200 nm. To probe the long-range variation of these domains, we make LFM measurements at locations separated by several microns on the ~5.2 nm thick flake of MoS_2 shown in the AFM image in Fig. 7(a). Figs. 7(b) and 7(c) show lateral trace images of the flake at the locations marked in fig. 7(a) by the solid red and blue dashed arrows, respectively. These two locations are separated by ~2.5 μ m on a single terrace and there are no step edges between them. Relative to the nearly vertically-oriented frictional features seen in Fig. 7(b), the frictional features seen in Fig. 7(c) are rotated ~ 55 °, indicating that the orientation is not maintained on the scale of several microns.

The stripes we observe seem to arise from the actual MoS₂ and do not appear to be due to any topographical distortions of its surface, such as microscopic corrugation, wrinkles, or ripples.[40-44] Although some of our contact mode LFM measurements have a very small striped topographical signal smaller than 0.05 nm in magnitude according to simultaneously acquired AFM height measurements, it seems likely that it is a measurement artifact due to a slight crosstalk from the lateral to vertical scanning probe signal resulting from slight misalignment of the PSD.[45] If the modulated friction signal were, instead, due to an actual height variation of the MoS₂ surface, we would expect the amplitude of the modulation to increase linearly with increasing load applied by the tip,[34] in contrast to the roughly constant behavior observed in Fig. 5(b). Another possible artifact that we exclude is poor feedback control, where a small surface corrugation could result in a modulated F_{net} if feedback were lost yielding a spatially varying F_f . However, such an extrinsic feedback issue seems inconsistent with our measurements, as it would likely depend on scan frequency, in contrast with the results in Fig. 4. Moreover, estimating the variation in the frictional force using a conservative upper-bound estimate of 0.05 nm as a possible height modulation (and assuming the measured 0.022 background friction coefficient and the 0.2 N/m vertical spring constant of the cantilever) gives a value two orders of magnitude smaller for the stripes than the signal we measure with LFM. Thus, we conclude that the measured modulated frictional stripes do not appear to directly stem from surface topographical variations.

Instead, our results suggest that the striped LFM signal is due to coupling of the asperity to a modulated strain on the surface of MoS_2 . Related to this possibility, it has recently been suggested that a single asperity tip could indirectly couple via a puckered region to strained graphene and its resulting ripples, leading to large (micron)

scale anisotropic friction.[20] Like the observed stripes we report here, that previous work did not see direct topographic evidence for any atomic- or nanoscale modulations. Instead, it was suggested that possible strain and ripples within the graphene are only detectable via a puckered region below an asperity as it slides over the surface.[20] The frictional stripes we observe seem to be consistent with a similar underlying mechanism, but with a much smaller (~4 nm) nanoscale modulation.

The stripe features we observe on MoS₂ also have intriguing similarities to recent reports of nanoscale modulated stick-slip of an asperity dragged over a graphite surface.[30, 31] As with our results here on MoS_2 , this previous work showed a modulation of the lateral signal along equally spaced parallel stripes with a length scale of approximately 4 nm. Like the report of anisotropic friction on graphene,[20] it was suggested that puckering underneath an asperity is a sensitive probe of local strain fields, which in the case of graphite results in nanoscale stick-slip events in an ordered arrangement of stripes.[30, 31] Similar to our report here and the anisotropic friction of Ref. [20], this strain field is only readily apparent through lateral force measurements, and not through direct topographical measurement. Although these are intriguing similarities between our observations and those on graphite, there is an important distinction between the two. In the case of MoS_2 , evidence for stick-slip events in the vicinity of the stripes is not apparent in the experiments, as it is for graphite.[30] Moreover, we also do not see any evidence of the associated scan speed dependence observed for the stripes in graphite related to stick-slip events,[31] suggesting that we are in fact observing a modulated friction at the 4 nm scale. The source of this difference could be due to the stiffer MoS₂ layers (in comparison to graphite) that do not allow for the puckered region under an asperity to be easily caught by the tip. Despite this lack of stick slip for MoS₂, the puckering underneath an asperity still appears in our measurements to be able to couple to the local strain resulting in a modulated friction. Overall, the surprisingly similar nanoscale spatial modulation of our lateral force and the previously reported striped stick-slip in graphite suggests there may be a common origin. Future experiments will need to determine if such nanoscale modulation is in fact a general property of such laminar materials and whether its properties can be suitably tuned for surface modification over extremely short length scales.

Conclusions

In conclusion, we have observed extremely small-scale mechanical properties of the laminar material MoS_2 within the 10 nm size regime. Low-adhesion asperities have been shown to catch and flex the MoS_2 edges over a length of order 10 nm. These MoS_2 edges are found to be 20% stiffer than those of graphene, with strain energies consistent with out-of-plane bending. In the vicinity of these MoS_2 edges, we have also obtained the first experimental evidence for a modulated striped nanoscale frictional phase. These frictional stripes are modulated on the extremely small 4.1 nm

length over domains greater than 100 nm. The ordered frictional stripes appear to be an intrinsic property of the MoS_2 surface layers, as the scanning properties of the asperity do not make a noticeable contribution to their modulation length. Our results suggest that the frictional stripes could be due to a modulation in the local strain in the MoS_2 that couples to the puckered region below a single asperity LFM tip. It is possible that these stripes are related to ripple strain that has been suggested as an underlying mechanism of electron scattering[41] and anisotropic friction in graphene.[20]

A particularly significant aspect of our results is the fact that the ~4 nm length scale of the modulated friction we observe has intriguing similarities to recently reported stick-slip lateral force stripes on graphite, which are also thought to arise from coupling puckered surfaces to nanoscale strain fields.[30, 31] In addition to having a very similar modulation length, our results and those of Refs. [30] and [31] only obtain a striped force modulation laterally, without appreciable topographic variation. This intriguing similarity raises the prospect that such ordered modulation of surface nanomechanical properties could be a general feature in the growing array of laminar materials. These results could have important implications to controlling and understanding the ultrashort nanomechanical features of MoS₂ that, due to its ubiquitous application as a dry-solid lubricant, is a promising material for future use as atomically-thin coatings to nanoscale machines. Moreover, while our work was under review we became aware of a recent manuscript posted on the arXiv[46] reporting observations of a striped phase on hexagonal BN (another laminar 2D material) and its relation to anisotropic friction which was attributed, alternatively, to surface adsorbates.[47-51] Taken together, this other work on BN and our results presented here on MoS_2 , it suggests the fascinating possibility that nanoscale frictional stripes might be widely observable in the growing family of 2D materials.

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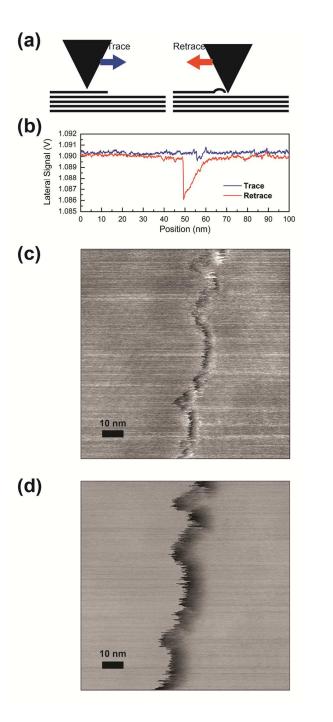
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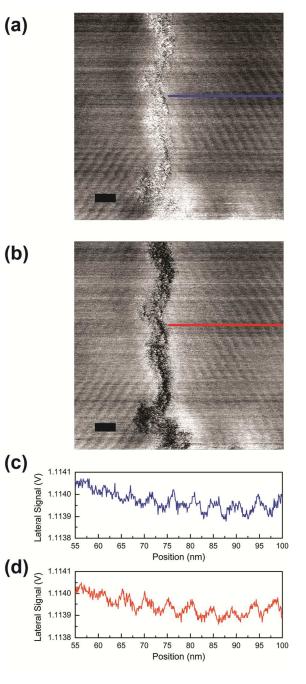


Fig. 1 LFM of a single layer MoS₂ step edge. (a) Schematic of round-trip LFM line scan taken over a single-layer step-edge of MoS₂. As illustrated by the schematic, the MoS₂ film has one extra layer on the left. (b) Round-trip LFM line scans where the blue step-down "trace" scan is from left to right and the red step up "retrace" scan is from right to left, consistent with the schematic in (a). LFM trace images over the same step edge are shown in (c) for the trace direction and (d) for the retrace direction. Measurements in (b)-(d) were made with a net load of F_{net} = 8.3 nN for a LFM tip with a 1.6 nN adhesion.

Fig. 2 Periodic frictional stripes. (a) LFM trace image of MoS₂ showing parallel frictional stripes. The MoS₂ on the right (left) side is ~6.5 nm (5.9 nm) thick (~10 (9) layers) as determined through AFM topography measurements. Upon crossing a step edge (the brighter vertical line) the angle of the stripes abruptly changes. (b) Same as (a), for the retrace direction. (c) and (d) are the average of 10 LFM scans at the locations specified by the blue and red lines in (a) and (b), respectively. These line scans show a 4.6 nm wavelength, which after correcting for the 27° angle between the line scan and the direction perpendicular to the oscillations gives a wavelength of 4.1 nm. Adhesion and F_{net} of the probe were 4.45 nN and 1 nN, respectively. Scale bars are 10 nm.

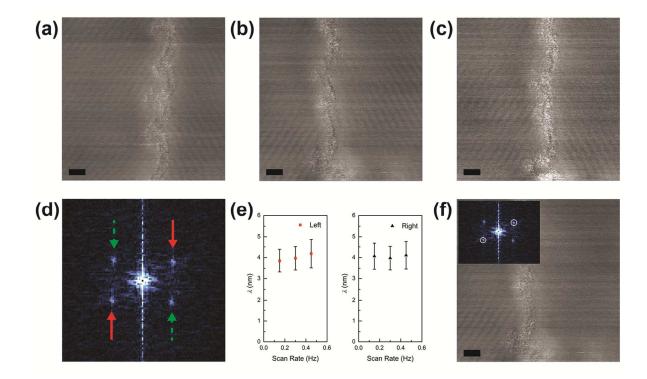


Fig. 3 (a)-(c) LFM trace images of the same MoS₂ region as in Fig. 2 taken at a range of scan speeds, with similar adhesions and net loads. (a) LFM trace with a scan speed of 0.15 Hz, adhesion of 4.35 nN, and F_{net} of 1.01 nN. (b) Image taken with a scan speed of 0.30 Hz, adhesion of 4.45 nN, and F_{net} of 1.0 nN. (c) Image taken with a scan speed of 0.45 Hz, adhesion of 4.45 nN, and F_{net} of 1.01 nN. (b) Image taken with a scan speed of 0.30 Hz, adhesion of 4.45 nN, and F_{net} of 1.0 nN. (c) Image taken with a scan speed of 0.45 Hz, adhesion of 4.13 nN, and F_{net} of 1.37 nN. (d) FFT of (b) with the peaks marked with the solid red (dashed green) arrows corresponding to the stripes on the right (left) side of the step edge (central bright vertical region) in (b). These peaks occur at wave number magnitudes of 0.252 nm⁻¹ and 0.253 nm⁻¹, corresponding to wavelength of 3.97 nm and 3.95 nm for the features on the right and left side, respectively. (e) Wavelength as a function of scan rate for features to the left (red squares) and right (black triangles) of the step edge, showing no dependence between scan rate and wavelength. (f) Selective removal of the circled peaks in the FFT (inset) with the resulting subsequent inverse transform showing that the associated stripes are removed from the image. All scales bars are 10 nm.

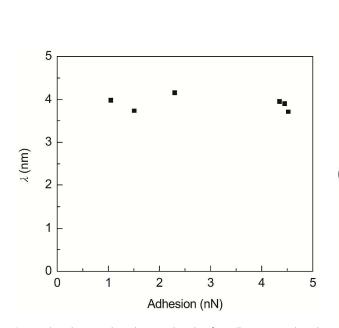


Fig. 4 Plot showing that the wavelength of oscillations is relatively independent of the adhesion of the LFM probes used. The wavelength is measured with tips having different adhesions.

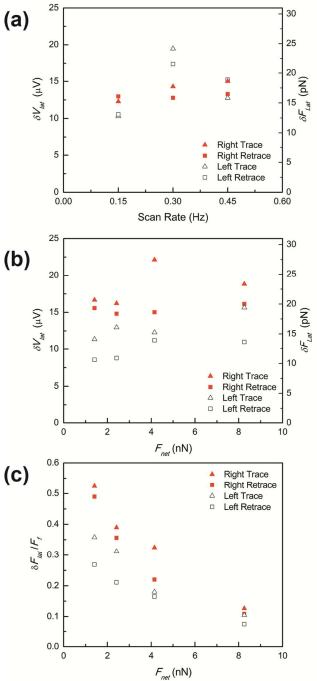


Fig. 5. Comparison of lateral force arising from the stripe features for various loads and scan rates with the same LFM probe. (a) Plot showing the RMS lateral voltage signal and corresponding lateral force of the stripe features as a function of scan rate, as determined through the FFT of the LFM signal. (b) Plot of the RMS lateral voltage and force arising from the stripes on the right (red) and left (black) side of the step edge as a function of F_{net} . For all four F_{net} values plotted, the same LFM probe was used, and the adhesion remained between 1.31 nN and 1.71 nN. (c) Plot of the ratio of the RMS lateral force due to the periodic stripes to the average frictional force, F_{f} , as a function of F_{net} .

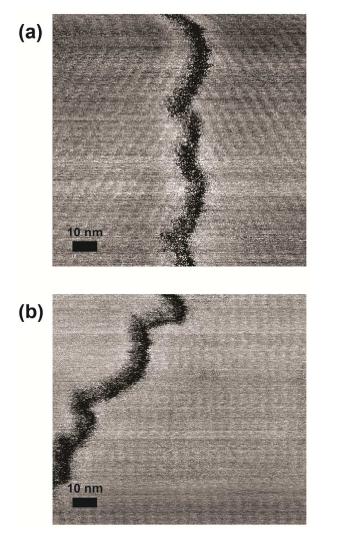
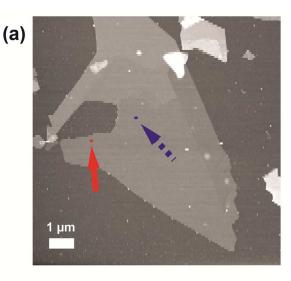


Fig. 6 LFM images showing the effects of varying the scan angle on the MoS₂ frictional features. (a) LFM retrace image with the LFM probe scanning perpendicular to the MoS₂ step edge, showing frictional features on both sides of the step edge. (b) LFM retrace after the sample was rotated ~30° clockwise. Frictional features on the right side of the step edge rotate with the sample and appear nearly perpendicular to the fast scan direction. The features on the left side of the step edge, which have been rotated so that they are closer to being parallel to the fast scan direction, are no longer visible. For reference, the red arrows in (a) and (b) mark the same point on the sample. Scale bars are 10 nm.



(b) 10 nm

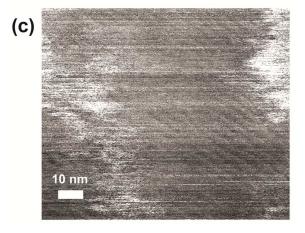
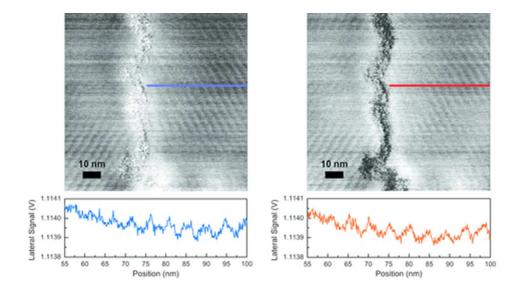


Fig. 7 AFM and LFM images showing frictional features at various locations on a MOS_2 flake. (a) Tapping mode AFM height image of a MOS_2 flake. (b) Lateral trace image taken at the location marked by the solid red arrow showing frictional features perpendicular to the fast scan direction. (c) Lateral trace image taken at the location marked by the dashed blue arrow. The frictional features at this location appear rotated ~55° relative to those in (b). The data in (a)–(c) were taken at the same scan angle, with the same probe.

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