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Superlubricity between MoS_2 Monolayers

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The ultralow friction between atomic layers of hexagonal MoS_2, an important solid lubricant and additive of lubricating oil, is thought to be responsible for its excellent lubricating performances. However, the quantitative frictional properties between MoS_2 atomic layers have not been directly tested in experiments due to the lack of conventional tools to characterize the frictional properties between 2D atomic layers. Herein, a versatile method for studying the frictional properties between atomic-layered materials is developed by combining the in situ scanning electron microscope technique with a Si nanowire force sensor, and the friction tests on the sliding between atomic-layered materials down to monolayers are reported. The friction tests on the sliding between incommensurate MoS_2 monolayers give a friction coefficient of $\approx 10^{-4}$ in the regime of superlubricity. The results provide the first direct experimental evidence for superlubricity between MoS_2 atomic layers and open a new route to investigate frictional properties of broad 2D materials.

Friction and wear induce a large portion of energy waste in mechanical systems and cause erosion.[1,2] Since putting lumbers under the giant stone when building the pyramid in ancient Egypt, humans have been studying friction and trying to find ways to reduce it for thousands of years. Hexagonal molybdenum disulfide (MoS_2) is a well-known lamellar compound that has been used as a solid lubricant and additive of lubricating oil for a long time.[3] It is generally recognized that the excellent performance of MoS_2 lubricant is attributed to the weak interaction and easy sliding between MoS_2 atomic layers.[4] In 1993 Martin et al. studied the atomic origin of the ultralow friction of a sputter-deposited MoS_2 coating in ultrahigh vacuum and attributed it to the superlubricity along incommensurate MoS_2 basal planes during intercrystallite slip.[5] Therefore, the frictional properties between MoS_2 atomic layers are important for the full understanding of the excellent performances of MoS_2 lubricant, and have been theoretically studied in several literatures.[6–8] It was found in theory that two incommensurate MoS_2 monolayers show extremely low friction due to the cancellation of atomic force in the sliding direction, while in commensurate case, the atomic forces were acted in the same direction, leading to 100 times larger friction than incommensurate situation.[6] The friction between two MoS_2 monolayers was also found to be reduced by imposing a perpendicular electric field to them.[7] Sliding motion of two commensurate MoS_2 monolayers was studied by means of first-principle calculations to determine their frictional properties with different load and orientation.[8] Despite the insightful theoretical studies, the frictional properties between 2D atomic layers of MoS_2 have not been directly tested in experiments due to the great challenges of such experiments.

In recent years, MoS_2 nanosheets with one or a few atomic layers have been successfully exfoliated from its bulk crystals or synthesized by chemical vapor deposition methods due to their great promise in nanoelectronics and catalyst applications.[9–14] It provides a good sample platform for studying the frictional properties between atomic layers of MoS_2. However, it is challenging to apply the conventional experimental methods in tribology to the friction tests between two atomic layers of MoS_2. For normal tribometers,[5,15,16] it is difficult to prepare atomic layers of MoS_2 coating on the both sliding sides of a tribometer and monitor their structural evolution during the test. Friction force microscope has been used as a powerful tool to study the frictional properties between a sharp tip and atomically thin sheets of lamellar materials including graphene and MoS_2,[17–20] and those between two thick stacks of graphene,[21] but it is still hard applied to the sliding between two atomic layers. Importantly, when a sharp tip slides on an atomically thin nanosheet, out-of-plane elastic deformation of the nanosheet will be induced, leading to an overestimate of friction.[17] In addition to MoS_2 nanosheets, a large library of 2D materials (e.g., graphene, boronitrene, phosphene, etc.) have been obtained and their van der Waals homostructures and heterostructures can be built through layer-by-layer stacking.[22,23] Due to weak van der Waals interaction, those vertical homostructures and heterostructures of 2D materials are expected to exhibit interesting frictional properties similar with those of MoS_2 nanosheets and deserve detailed studies in experiments. Consequently, an experimental method meeting the requirement of those studies is highly desired, but still unavailable.

In this paper, a versatile method for studying the frictional properties between atomic layers of 2D materials is developed by combining the in situ scanning electron microscope (SEM) technique with the friction force sensor of a Si nanowire cantilever. The friction tests based on the sliding of a single-layer

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DOI: 10.1002/adma.201701474

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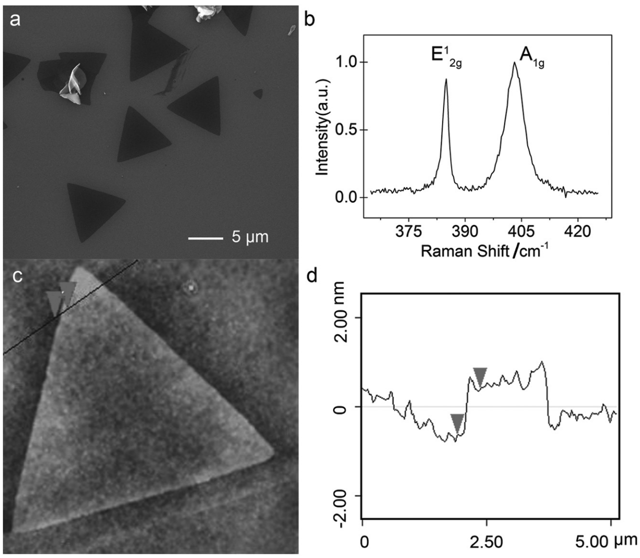

Figure 1. a) SEM image of MoS_2 nanosheets grown on SiO_2/Si substrate. b) Raman spectrum of a MoS_2 nanosheet. c) Atomic force microscopic image of a triangle MoS_2 nanosheet. d) Sectional profile of a triangle MoS_2 nanosheet along the solid line in (c).

MoS_2 nanoflake on another incommensurate single-layer MoS_2 nanosheet on substrate give a friction coefficient in the order of 10^{-4}, indicating the superlubricity between MoS_2 monolayers. Our work provides the first direct experimental evidence for superlubricity between atomic layers of hexagonal MoS_2 and a general method for studying the frictional properties of 2D materials. The friction tests between MoS_2 monolayers also provide the first friction tests between atomic-layered materials down to the extreme in thickness.

Friction tests in this work are in situ performed inside a SEM equipped with a nanomanipulator, so the whole test process can be monitored in real time via SEM observation.[24,25] **Figure 1**a shows a SEM image of MoS_2 nanosheets studied in this work. MoS_2 nanosheets are grown on SiO_2/Si substrate and in isosceles triangle shape with a side length of ≈10 µm. Raman spectrum of our studied MoS_2 nanosheets with an incident laser wavelength of 532 nm is shown in Figure 1b. The position difference of 18.1 cm^{-1} between E^1_{2g} peak (385.0 cm^{-1}) and A_{1g} peak (403.1 cm^{-1}) indicates that the MoS_2 nanosheets just have single layer in thickness.[26] The single-layer character is further confirmed by atomic force microscopic images shown in Figure 1c,d, where the thickness of MoS_2 nanosheets is measured to be less than 1 nm (0.97 nm).[19]

Before performing friction tests on two sliding single-layer MoS_2 nanosheets, two problems need to be solved. The first one is how to achieve the sliding between two single-layer MoS_2 nanosheets and the second one is how to measure the friction force during the sliding. In order to overcome the challenges, an experimental setup as shown in **Figure 2**a is adopted. In the setup, a Si nanowire is singly clamped to a tungsten (W) tip that can move in three independent directions at a step size of a few nanometers under the control of a nanomanipulator. The Si nanowire is first used as a nanoprobe to peel off a single-layer MoS_2 nanoflake from a triangular nanosheet, and to place the nanoflake on another single-layer MoS_2 nanosheet on

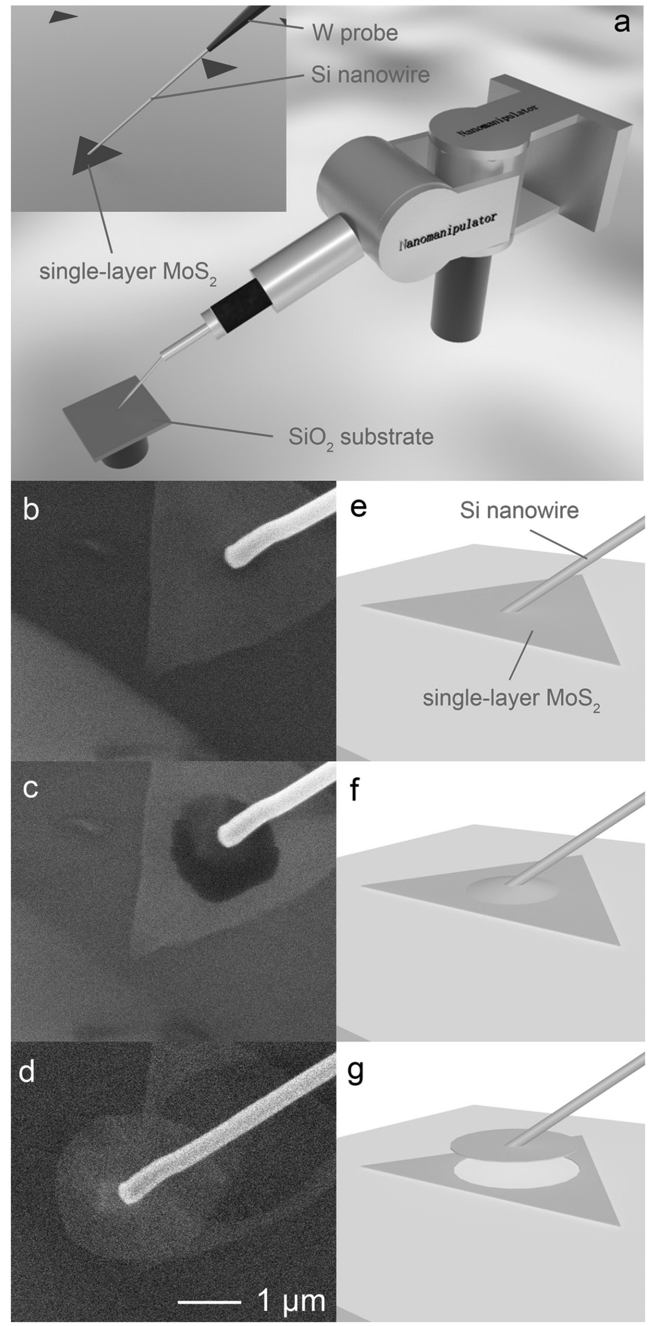

Figure 2. a) Schematic diagram of the experimental setup for friction tests. The inset is an enlarged view of the free end of W tip. b–d) SEM images showing the process of peeling off a single-layer MoS_2 nanoflake from SiO_2/Si substrate by using a Si nanowire probe. e–g) Schematic drawings of the peeling process shown in (b–d).

substrate for sliding. Then, the Si nanowire is used as a cantilever force sensor to detect the friction force during the sliding. The force detected by a cantilever sensor is proportional to its deflection that can be directly measured by SEM imaging. The spring constant of a Si nanowire cantilever is determined as $k = \frac{3\pi r^4}{4L^3}E$, where r and L are ,respectively, the radius and length of Si nanowire measured directly by SEM imaging, E

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is Young's modulus.[27] Young's modulus of Si nanowires used in this work was measured to be 134 ± 28 GPa by an in situ electric-induced resonance method (see Figure S1 in the Supporting Information).[28]

The peeling of a single-layer MoS_2 nanoflake from a MoS_2 nanosheet on SiO_2/Si substrate by using a Si nanowire probe is shown in Figures 2b–g. The free end of Si nanowire probe is first manipulated to make contact with a MoS_2 nanosheet grown on substrate at its center, then the contact is irradiated by a focused electron beam of SEM for tens of seconds to enhance its mechanical strength by electron beam induced amorphous carbon deposition (Figure 2b,e).[24,29] After MoS_2 nanosheet is firmly adhered to the Si nanowire probe, the latter is lift under the control of the nanomanipulator to peel MoS_2 nanosheet off the substrate. It can be seen from Figure 2c that, when MoS_2 nanosheet is separated from substrate, its secondary electron imaging contrast exhibits remarkable change to show a dark contrast. With the lifting of Si nanowire, a round area in dark contrast becomes larger and larger, indicating that larger and larger region of the MoS_2 nanosheet is separated from substrate. When the strain of MoS_2 nanosheet reaches its elastic limit, it fractures. Then, a single-layer MoS_2 nanoflake is successfully peeled off from the substrate and adhered to the free end of the Si nanowire probe in an orientation parallel to substrate (Figure 2d,g). It is worth noting that, even though the upper surface of the nanoflake could be contaminated by electron beam induced amorphous carbon, the bottom surface that will be used for friction test is expected to be free of contaminations.

After a single-layer nanoflake adhered to the free end of a Si nanowire probe is obtained, the frictional properties between single-layer MoS_2 nanosheets are immediately tested as schematically shown in **Figure 3**a. The nanoflake is first transferred to be above another piece of triangular MoS_2 nanosheet grown on substrate (Figure 3b), then is lowered to make contact with the latter. Once making contact with the nanosheet on substrate, the secondary electron imaging contrast of nanoflake becomes as dark as that of MoS_2 nanosheet on substrate (Figure 3c), which provides a good guide for judging the contact area between a nanoflake and a nanosheet substrate. To make the nanoflake slide on a nanosheet substrate, the W tip is manipulated to move in horizontal direction and perpendicular to the axis of Si nanowire cantilever. Due to the presence of friction force, MoS_2 nanoflake does not move together with the W tip at the beginning, which causes the deflection of Si nanowire cantilever. With the increase of cantilever deflection, the static friction of MoS_2 nanoflake is balanced by the bending force of cantilever, and MoS_2 nanoflake starts to slide on MoS_2 substrate (Figure 3d and Video S1 (Supporting Information)). In this case, the friction force during the sliding can be measured from the bending force of Si nanowire cantilever.

The Si nanowire cantilever in Figure 3d is 126 nm wide and 5.28 μm long, which gives a spring constant of 0.034 N m^{-1} according to the above formula. The deflection (δ) of the cantilever was measured to be 1.07 μm during sliding. A small deflection-to-length ration of 0.2 (corresponding to a deflection angle of ≈11°) ensures that the deflection of the cantilever is in a linear range and Hooke's law ($F = k\delta$) can be applied. So a friction force (F) of 36.1 nN is obtained for the nanoflake. In

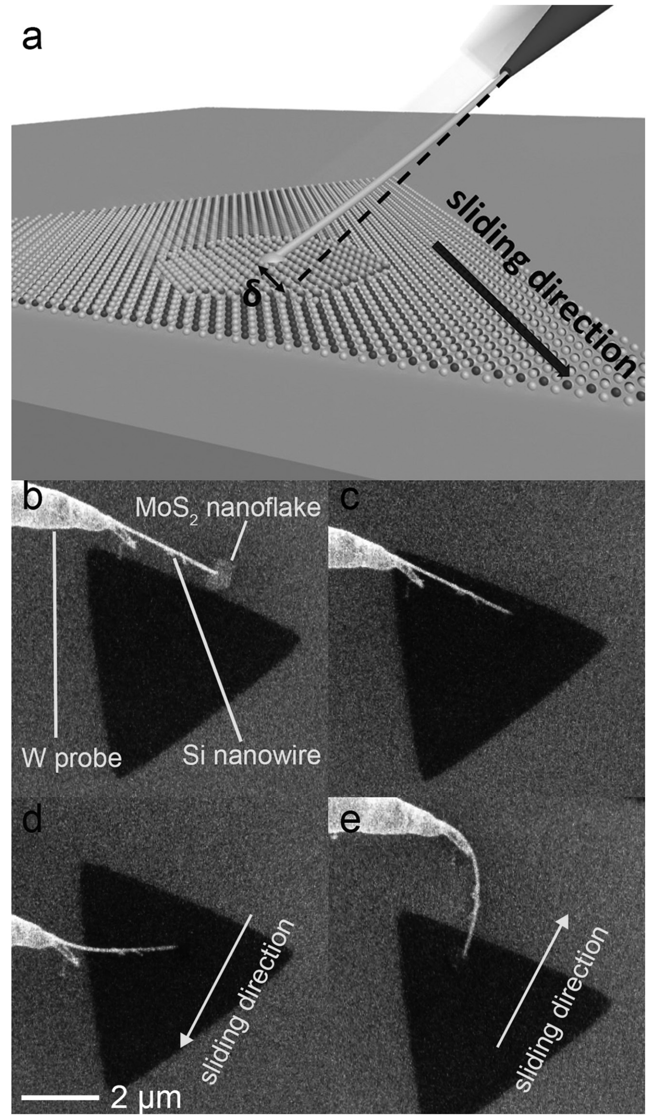

Figure 3. a) Schematic drawing of the friction test on the sliding of a single-layer MoS_2 nanoflake on a single-layer MoS_2 nanosheet substrate. b–e) SEM images showing the process of the friction test. A single-layer MoS_2 nanoflake adhered to Si nanowire probe is first transferred to be above a single-layer MoS_2 nanosheet on substrate (b), then is lowered to make contact with the latter (c). Both low friction (d) and high friction (e) states are observed when a MoS_2 nanoflake is manipulated to slide on a MoS_2 substrate.

our experiments, the deflection of Si nanowire cantilever perpendicular to the sliding direction was carefully avoided, so the compressive force applied by Si nanowire cantilever to nanoflake was negligible. Normal load of a nanoflake is mainly contributed by the cohesive force between the nanoflake and MoS_2 substrate. The cohesive force can be estimated by $N = \sigma S/c$ with $\sigma = 0.14$ J m^{-2} being the interface energy of MoS_2,[30,31] $c = 0.3$ nm being the interaction length between MoS_2 atomic layers,[4] and $S = 0.6$ μm^2 being the contact area between the nanoflake and MoS_2 substrate. It should be noted that, the contact area is sometimes smaller than the area of nanoflake since the curling of the edge may take place for some nanoflakes.

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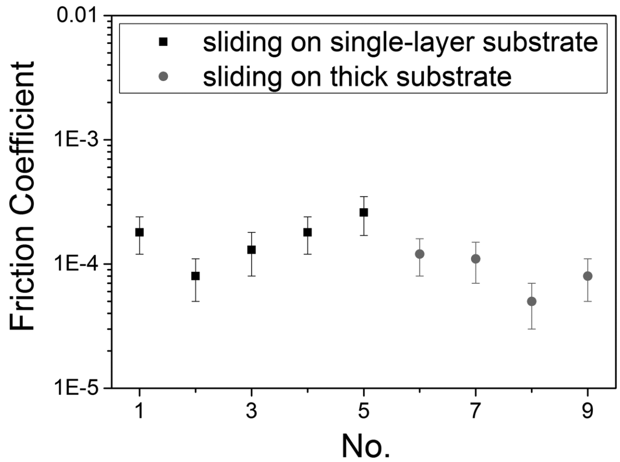

Figure 4. Friction coefficient of the sliding of a single-layer MoS_2 nanoflake on both single-layer MoS_2 substrate and thick MoS_2 substrate.

In this case, the contact area is determined by the area exhibiting dark contrast for the reason mentioned above. A cohesive force of 280 μN is obtained for the nanoflake in Figure 3d, so a friction coefficient ($\mu = F/N$) of 1.3×10^{-4} is obtained. The ultralow friction coefficient further confirms the negligible compressive force applied by Si nanowire cantilever when compared to the cohesive force, since the former, if it presents, is comparable to the friction force. Our measured friction coefficient may be underestimated by the ripple of nanoflake since it makes the contacting area overestimated. However, single-layer MoS_2 nanosheets can follow conformally the topography of the substrate,[19] so the ripple of MoS_2 nanoflakes lying on an atomically flat MoS_2 substrate is negligible. In contrast to the ultralow friction coefficient between MoS_2 monolayers, the friction coefficient between a Si nanowire and a MoS_2 monolayer is measured to be 0.42, three orders of magnitude larger than the former (see Figure S2 in the Supporting Information).

In total, five different single-layer nanoflakes were tested and their friction coefficients are shown in **Figure 4**. A summary of friction tests performed in this work is shown in **Table 1**. It can be seen that friction coefficient of a single-layer MoS_2 nanoflake sliding on a single-layer MoS_2 nanosheet substrate is in the range of 0.8×10^{-4}–2.6×10^{-4}. According to the definition of superlubricity ($\mu < 10^{-3}$),[2,18] the tested friction between two MoS_2 monolayers is in the regime of superlubricity. Our measurement results agree well with the ultralow friction coefficient (in 10^{-3} range) of sputter-deposited MoS_2 coating measured by an ultrahigh vacuum tribometer.[5] Based on the observation of twisted basal planes in wear particles by high-resolution transmission electron microscopy, the ultralow friction coefficient of the MoS_2 coating was attributed to the superlubricity between incommensurate basal planes of hexagonal MoS_2.[5] However, no direct test on the frictional properties between basal planes was carried out and a direct experimental evidence for the superlubricity between incommensurate basal planes is still absent up to date. Herein, our measured ultralow friction coefficient between two MoS_2 monolayers gives the first direct experimental evidence for the superlubricity between basal planes of hexagonal MoS_2, and a strong support for the hypothesis that the excellent performances of MoS_2 lubricant is attributed to the ultralow friction between MoS_2 atomic layers.

The ultralow friction coefficient indicates that the nanoflakes tested in our experiments formed an incommensurate interface with single-layer MoS_2 nanosheet substrate. Theoretical studies of frictional properties between two MoS_2 monolayers showed that an incommensurate interface with extremely low friction is formed when the misfit angle between two nanosheets is in the range of 5°–55° and a commensurate interface with a friction of 100 times larger is formed when the misfit angle is close to 0° and 60°.[6] By comparing the angles formed between Si nanowire and the edges of triangular MoS_2 nanosheets during peeling and sliding, the misfit angles between MoS_2 nanoflake and nanosheet substrate during friction tests were successfully measured to be 24.8°, 35.4°, and 32.2° for three samples (see Figure S3 in the Supporting Information), in good agreement with the theoretical prediction. It is worth noting that it is difficult to accurately control the misfit angle between nanoflake and substrate during sliding, because the misfit angle changes with the deflection of Si nanowire cantilever due to the rigid connection between nanoflake and cantilever.

Few nanoflakes were also observed to experience much larger friction than those in Figure 4. Figure 3e shows the sliding of the same nanoflake as that in Figure 3d, but with a different misfit angle between the nanoflake and substrate. It can be seen that the Si nanowire cantilever exhibited a much

Table 1. A summary of friction tests performed in this work, including sliding on both single-layer (sample SL) and thick-layer (sample TL) MoS_2 substrate. δ, r, L are the deflection, radius, and length of Si nanowire cantilever. S is the contacting area between MoS_2 nanoflake and MoS_2 substrate. F is friction force. μ is friction coefficient. θ is misfit angle between MoS_2 nanoflake and MoS_2 substrate.

Sample ID	δ [μm]	r [nm]	L [μm]	S [μm^2]	F [nN]	μ [10^{-4}]	θ [°]
SL1	4.37 ± 0.08	217 ± 2	16.69 ± 0.58	7.90 ± 0.40	658.6 ± 214.0	1.8 ± 0.6	
SL2	3.28 ± 0.18	284 ± 2	40.83 ± 1.43	2.63 ± 0.13	99.1 ± 35.2	0.8 ± 0.3	
SL3	1.07 ± 0.03	63 ± 2	5.28 ± 0.19	0.60 ± 0.03	36.1 ± 12.8	1.3 ± 0.5	24.8 ± 1.0
SL4	1.67 ± 0.04	151 ± 2	12.76 ± 0.45	1.60 ± 0.08	131.7 ± 42.8	1.8 ± 0.6	35.4 ± 1.0
SL5	0.06 ± 0.01	110 ± 2	3.89 ± 0.14	0.36 ± 0.02	43.3 ± 15.8	2.6 ± 0.9	32.2 ± 1.0
TL1	0.79 ± 0.01	73 ± 2	7.54 ± 0.26	0.30 ± 0.02	16.5 ± 5.5	1.2 ± 0.4	
TL2	3.68 ± 0.08	70 ± 2	12.26 ± 0.43	0.31 ± 0.02	15.1 ± 5.2	1.1 ± 0.4	
TL3	1.02 ± 0.02	70 ± 2	10.12 ± 0.35	0.32 ± 0.02	7.4 ± 2.6	0.5 ± 0.2	
TL4	0.21 ± 0.01	90 ± 2	6.04 ± 0.21	0.55 ± 0.03	19.7 ± 6.4	0.8 ± 0.3	

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larger deflection (≈3.6 μm) than that in Figure 3d (see Video S2 in the Supporting Information). The deflection was so large that the nanoflake slid in a direction nearly parallel to the axis of Si nanowire. Moreover, the nanoflake was partly separated from substrate under the drag of Si nanowire as judged from its brighter contrast compared to that in Figure 3d. Even though in this case the friction force and friction coefficient cannot be quantitatively determined due to nonlinear deformation of Si nanowire cantilever, the much larger deflection of Si nanowire cantilever and smaller contacting area during the sliding indicate that the friction coefficient is much larger than that in Figure 3d. Due to the large friction, the nanoflake was seriously twisted and folded under the drag of Si nanowire, which makes it difficult to determine the misfit angle. A commensurate interface is expected to form between the nanoflake and MoS_2 substrate in Figure 3e and be responsible for the large friction.[6]

Frictional characteristics between atomically thin MoS_2 sheets and a sharp silicon tip studied by frictional force microscopy indicated that friction force increased with the decrease of sheet thickness,[17] which was attributed to the increased susceptibility of thinner sheets to out-of-plane elastic deformation caused by a sliding silicon tip. To figure out whether the effect exists in our experiments or not, the frictional properties of single-layer MoS_2 nanoflakes sliding on a thick MoS_2 substrate is also studied in a similar way (see Figures S4 and S5 in the Supporting Information). Thick MoS_2 sheets used as sliding substrate were prepared by mechanical exfoliation method and are estimated to be tens of nanometers in thickness. In total, four nanoflakes sliding on thick MoS_2 substrate were tested and their friction coefficients are also shown in Figure 4. It can be seen that nanoflakes sliding on thick MoS_2 substrate have quite similar friction coefficient (in the range of 0.5×10^{-4}–1.2×10^{-4}) with that of nanoflakes sliding on single-layer MoS_2 substrate, indicating that our measured friction coefficient shows no obvious dependence on the thickness of MoS_2 substrate. So single-layer MoS_2 substrate is expected to exhibit negligible out-of-plane elastic deformation during nanoflake sliding.

Even though friction tests were in situ performed inside a high vacuum SEM with a chamber vacuum of $\approx 10^{-4}$ Pa, the measured friction coefficient may still deviate from the theoretical value for ideal incommensurate interface between MoS_2 atomic layers due to the influence of some undesired factors. It was reported that friction coefficient of sputter-deposited MoS_2 coating strongly depends on the environmental pressure and is sensitive to the contamination of oxygen, carbon, and water.[5,32] In our experiments, MoS_2 nanosheets were exposed in air and no degassing process was performed before tests in SEM. So, some oxygen and water molecules may be adsorbed on MoS_2 nanosheets during tests and increase friction coefficient. The pucker and dangling bonds at the edge of nanoflake may increase friction coefficient as well.[17,31] In this case, friction coefficient will depend on the area of nanoflake since the effect of the pucker and dangling bonds at the edge is more remarkable for the nanoflakes with smaller area. Moreover, the effects of electron beam irradiation on friction coefficient may be not negligible, since our friction tests were performed under SEM observation. Amorphous carbon deposition induced by electron beam irradiation was observed to increase the friction force between two sliding graphite microflakes.[31,33] Therefore, the friction coefficient of a perfect incommensurate interface formed by two ideal MoS_2 atomic layers is thought to be even smaller than that measured in this work due to above reasons, and it is certainly in the regime of superlubricity.

The method developed here can be applied to other 2D materials (e.g., graphene, boronitrene, phosphene, etc.) as long as the atomic nanosheets of 2D materials lying on substrate can be obtained. In addition to the frictional properties between atomic layers of the same kind of 2D materials, it can be applied to study the frictional properties between atomic layers of different 2D materials. For such friction tests, more than one kind of 2D materials are put on SEM sample holder. After a nanoflake of one kind 2D material is peeled off from substrate, it is then manipulated to slide on the nanosheet of another 2D material and frictional properties between heterostructures of 2D materials can be tested. More importantly, in contrast to friction tests using tribometers and friction force microscopes, the whole process of friction tests can be monitored in real time under the observation of SEM, which unambiguously clarifies what happens during friction tests. The method can also be applied to investigate the bonding energy between the atomic nanosheets of 2D materials and a substrate when the force of peeling the nanosheet off the substrate is quantitatively measured. If the substrate is an atomic nanosheet of 2D materials as in this work, the interlayer bonding energy between homostructures and heterostructures of 2D materials can be determined by the method.

In summary, a versatile method for friction tests between atomic layers of 2D materials is developed and a direct experimental evidence for the superlubricity between atomic layers of hexagonal MoS_2 is reported for the first time. Friction tests on the sliding of a single-layer MoS_2 nanoflake on another incommensurate MoS_2 substrate give an ultralow friction coefficient of $\approx 10^{-4}$ in the regime of superlubricity. The friction coefficient is found to exhibit no dependence on the thickness of MoS_2 substrate down to single atomic layer. The superlubricity between MoS_2 monolayers indicates that superlubricity between incommensurate atomic-layered materials can preserve, even their thickness is scaled down to the extreme of monolayer. Our results suggest that atomic-layered MoS_2 can be a good candidate for lubricating coating at atomic interfaces.

Experimental Section

The experiments were in situ carried out at room temperature inside a high-vacuum SEM (FEI Quanta 600F) with a nanomanipulator (Kleindiek MM3A) installed in the SEM chamber. Si nanowires used in this work were grown on a Si wafer by chemical vapor deposition method and had good crystalline structure with uniform diameter along nanowire axis.[34,35] To construct a Si nanowire cantilever, a W tip was manipulated to make contact with a Si nanowire protruding from the edge of Si wafer, and the orientation of the Si nanowire was carefully adjusted to make it align well with W tip. The mechanical strength of the contact between Si nanowire and W tip was enhanced by depositing amorphous carbon induced by electron beam irradiation.[29] After the Si nanowire was detached from Si wafer by retracting W tip, a Si nanowire cantilever was successfully prepared. The length of Si nanowire cantilevers was carefully determined by SEM imaging after they were adjusted to be horizontal. Single-layer MoS_2 nanosheets were grown by chemical vapor

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deposition method on SiO_2/Si substrate using MoO_3 (≥99.5%, Sigma-Aldrich) and S powder (≥99.5%, Sigma-Aldrich) as precursors, under ambient pressure of ultrahigh purity Argon flowing at 775 °C.[11]

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Acknowledgements

This work was supported by the National Basic Research Program of China (Grant Nos. 2013CB933604, 2016YFA0300903), the NSF of China (Grant Nos. 61371001, 61621061, 11474006, 51522201).

Conflict of Interest

The authors declare no conflict of interest.

Keywords

2D materials, friction properties, in situ electron microscopy, MoS_2, superlubricity

Received: March 15, 2017
Revised: April 3, 2017
Published online:

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