Nanotribology of SiP nanosheets: Effect of thickness and sliding velocity

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Abstract: Two-dimensional compounds combining group IV A element and group V A element were determined to integrate the advantages of the two groups. As a typical 2D group IV–V material, SiP has been widely used in photodetection and photocatalysis due to its high carrier mobility, appropriate bandgap, high thermal stability, and low interlayer cleavage energy. However, its adhesion and friction properties have not been extensively grasped. Here, large-size and high-quality SiP crystals were obtained by using the flux method. SiP nanosheets were prepared by using mechanical exfoliation. The layer-dependent and velocity-dependent nanotribological properties of SiP nanosheets were systematically investigated. The results indicate the friction force of SiP nanosheets decreases with the increase in layer number and reaches saturation after five layers. The coefficient of friction of multilayer SiP is 0.018. The mean friction force, frictional vibrations, and the friction strengthening effect can be affected by sliding velocity. Specially, the mean friction force increases with the logarithm of sliding velocity at nm/s scale, which is dominated by atomic stick-slip. The influence of frequency on frictional vibration is greater than speed due to the different influences on the change in contact quality. The friction strengthening saturation distance increases with the increase in speed for thick SiP nanosheets. These results provide an approach for manipulating the nanofriction properties of SiP and serve as a theoretical basis for the application of SiP in solid lubrication and microelectromechanical systems.

Keywords: SiP nanosheet; 2D material; atomic force microscopy (AFM); nanotribology

1 Introduction

More and more micro-electro-mechanical systems and nano-electro-mechanical systems have been widely used in integrated sensors, aerospace applications, and intelligent control systems with the development of nanotechnology [1]. However, as the size of devices shrinks from macroscales to micro/nanoscales, the sharply increased specific surface area has brought serious scale effects and surface/interface problems [2]. The body forces become insignificant compared to the surface forces including friction force and adhesion force, making high coefficient of friction and excessive wear rate become the main factor affecting the service life and reliability of micro/nano devices [3]. In this situation, some friction law in macroscales no longer works. Hence, studying the nanotribological properties of materials and exploring the origin of friction from the atomic scale can provide theoretical guidance for reducing friction and wear. This process is of vital
importance for improving the performance of micro/nano devices and efficient use of energy.

A large number of lubricating materials with excellent lubricating effect have been developed to overcome the friction and wear problems of micro/nano devices [4]. Among these materials, 2D materials with nanoscale ultrathin crystal structure, outstanding mechanical properties, low interlayer shear force, and high adhesion have become the most widely studied and applied materials in nanotribology [5]. As the first 2D material discovered by mankind, graphene has been extensively studied in the field of nanotribology [6, 7]. However, as a lubricant, graphene still has some certain problems. Graphene is easy to suspend on the surface of the substrate with large surface roughness, severely weakening the lubrication effect [8]. Graphene is prone to wrinkles owing to its ultrathin monoatomic layer structure. The anti-friction and anti-wear properties are seriously reduced. As a typical representative of transitional metal dichalcogenides, MoS2 exhibits excellent lubrication performance [9, 10]. The moisture-sensitive characteristics and the resulting sharp decrease in lubrication performance hinder its wide application in many conditions [11, 12]. Black phosphorus (BP) has received great attention in the field of optoelectronics due to its excellent properties, such as wide adjustable band gap and high carrier mobility [13, 14]. Other researchers have investigated its friction properties and found that it has excellent lubricating properties. BP is easily decomposed under the coexistence of air, water molecules, and visible light, resulting in instability of its tribological properties [15]. Therefore, exploring new 2D materials with more stable and better lubricating properties is of great importance.

In recent years, SiP, a newly discovered 2D compound combining IV A element and VA element has attracted the attention of researchers [16–19]. From the perspective of element composition and structural characteristics, SiP may combine the advantages of the two groups and compensate for their disadvantages at the same time [20–23]. Relevant research shows that the pyrolysis temperature of SiP is 1,045 °C [24], indicating its high thermal stability. The cleavage energy of SiP is 0.26 J/m², which is lower than that of graphene (0.37 J/m²), as obtained from experimental test. This condition shows that SiP may be more prone to interlayer sliding and may have better lubricating properties than graphene [16, 25]. Therefore, SiP has great potential to be applied in lubrication. However, the fundamental nanofriction properties of SiP have not been studied experimentally. The basic tribological information of SiP can enrich the nanotribology database of 2D materials and promote the development of tribo-informatics [26]. Studying the nanofriction properties and regulation mechanism of SiP is of great importance.

Here, large-size and high-quality bulk SiP single crystals were obtained by using flux method. SiP nanosheets with different layers were fabricated by using mechanical exfoliation. The basic structural and fundamental physical properties of SiP were characterized. The layer-dependent and velocity-dependent friction properties of SiP nanosheets were systematically investigated through atomic force microscopy (AFM). The influence of speed and frequency on frictional vibration was analyzed in detail. The influence mechanism of speed on SiP friction strengthening effect was studied. The results show that the nanofriction of SiP can be controlled in terms of layer thickness. The average friction force, friction vibration, and friction strengthening effect in nanofriction can be affected by sliding speed.

2 Experimental

2.1 Preparation of SiP nanosheets

SiP bulk crystals were obtained by using flux growth method. The schematic of the pit furnace used for crystal growth is shown in Fig. 1(a). Silicon (Si) and red phosphorus (P) as the starting materials and tin (Sn) as the flux were used for the growth of SiP. The molar ratio of the starting reactants is Si:P:Sn = 1:1:5. The growth temperature is around 1,150–1,200 °C. SiP crystals were obtained after cooling the furnace at a rate of 0.5 °C·h⁻¹. N-doped Si covered with dry oxidation generating 300 nm-thick SiO₂ was used to prepare weakly bound SiO₂/Si substrates. The substrate was sonicated in acetone, ethanol, and deionized water successively for 10 min and then dried with nitrogen. SiP nanosheets were mechanically exfoliated from
SiP bulk crystals and deposited on cleaned SiO₂/Si substrates. The identification and position of SiP nanosheets were determined through optical microscopy (SZX7).

2.2 Characterization of SiP nanosheets

Some of the prepared SiP nanosheets with desired thickness were selected for Raman experiments. Raman spectra analyses were conducted by using an HR 800 system (Horiba Jobin Yvon). The sample was excited with a 532 nm laser, and the lateral resolution was 500 nm. The chemical composition information of SiP nanosheets was obtained through X-ray photoelectron spectroscopy (XPS) and using Al Kα excitation radiation. The main core level photoemission spectra of Si and P were taken. The binding energy of C 1s at 284.8 eV from adventitious carbon was taken as a reference. The surface morphologies of SiP were observed with a field emission scanning electron microscope (FE-SEM, JSM-7610F, JEOL).

2.3 Friction experiment of SiP nanosheets

A multimode 8 (Bruker) AFM system was used under lateral force mode (LFM) to obtain the topography and friction data simultaneously. SiP nanosheets with 1, 2, 5, 7, 13, and other layers were fabricated on the silicon substrate. A nanosensors PPP-LFMR cantilever with a spring constant of 0.01–1.87 N/m and a calibration torsional constant of 5.813 nN/V (wedge calibration method [27]) was used. The applied load was set by changing the deflection setpoint and the calibration normal constant (thermal noise method) is 13.22 nN/V. More tip parameters details can be found in Table. S1 in the Electronic Supplementary Material (ESM). Before collecting nanofriction experiment data, we will pre-rub the new tip for a period of time to...
ensure that the conditions of experiment remain relatively stable. Figure S1 in the ESM is the SEM image of adopted tip after nanofriction experiment. The AFM tip slid on different nanosheets back and forth with a sliding distance of 20 nm under a series of scanning rates. The friction data were acquired during sliding, and the quantitative friction was based on the average of values measured by repeating friction loops five times on the same sample. As shown in Fig. S2 in the ESM, no obvious wear was found in the experimental area before and after nanofriction experiment. Therefore, it can be considered that the nanofriction experiment has negligible influence on the wear/material removal of the SiP nanosheets surface. All frictional experiments were performed under ambient conditions (20–30 °C and 30%–40% relative humidity).

3 Results and discussion

3.1 Characterization of SiP nanosheets

The SiP bulk crystals were prepared by using flux growth method on a pit furnace, as shown in Fig. 1(a). The prepared large-sized SiP bulk crystals in our experiment are shown in the inset in Fig. 1(c). The SiP nanosheets of the subsequent experiments were obtained from these crystals by using mechanical exfoliation. The crystallographic crystal structure of SiP with an obviously layered structure along the c-axis is shown in Fig. 1(b). The interlayer distance of SiP is 6.8 Å. Single-layer SiP has five-membered ring and hexatomic ring structure. SEM and EDS were performed to determine the layered characteristic and element distribution of SiP. The SEM images shown in Figs. 1(c) and 1(d) indicated that the SiP nanosheets have obvious layered characteristic and a crack-free surface. The corresponding elemental maps with even distribution indicated the high quality of SiP nanosheets, as shown in Figs. S3(a) and S3(b) in the ESM.

The obtained SiP single crystal was also characterized through XRD, as shown in Fig. 1(e). The calculated value of XRD is derived from the Diamond software using the crystallographic data (in CIF format) for SiP crystal. The derived XRD data contains the diffraction peaks of all theoretical crystal faces of SiP crystal. While for SiP nanosheets, two obvious diffraction peaks located at 13.24° and 26.28° arise from the diffraction of (002) and (004) planes, indicating that the natural growth surface of SiP is the crystallographic ab-plane. The well-defined and narrow diffraction peaks indicated that the obtained SiP single crystals are in single phase and high quality. Surface analysis of SiP crystals is conducted through XPS to determine the binding energy of SiP, as shown in Fig. 1(f). The O 1s peak is considerably small, indicating that the SiP crystals sample has a small degree of oxidation during the test. The XPS pattern for Si 2p and P 2p is shown in Figs. S3(c) and S3(d) in the ESM. Two peaks located at 100.87 and 101.53 eV are assigned to Si 2p3/2 and 2p1/2 respectively, indicating different chemical environments, Si–Si and Si–P. The P 2p spectrum with two peaks located at 129.85 and 130.70 eV is fitted by considering two resolved peaks [28].

The schematic of the experimental AFM for SiP nanofriction experiment is shown in Fig. 2(a). The surface topography of the as-prepared multi-layer SiP nanosheets on the Si/SiO2 substrate is shown in Fig. 2(b). The Optical image of SiP nanosheets with different layers on SiO2 substrate can be seen in Fig. S3(e) in the ESM. Figure 2(c) is the detailed display of SiP layered texture in the red box in Fig. 2(b), which further shows that the SiP nanosheets obtained by using mechanical exfoliation have obvious layered characteristic. The surface topography of single-layer SiP and few-layer SiP nanosheets is shown in Fig. 2(d). The height profile corresponding to the white dashed line is shown in Fig. 2(e). From this height profile, the thicknesses of the two areas of SiP in Fig. 2(d) are approximately 1.13 and 6.2 nm, indicating that the corresponding layer number is approximately 1 and 9, respectively. The thickness of single-layer SiP measured by AFM is larger than the theoretical value due to the experimental instrument error and the contact interface gap. The Raman spectra of SiP nanosheets with different layers are shown in Fig. 2(f). Three Raman peaks with sufficient intensities can be identified at 261, 305, and 465 cm⁻¹, and correspond to A1₆, A₁₇, and A₂₆ phonon modes by comparing the experimental data and calculated data [18]. The intensity of these peaks gradually decreases with the decrease in the number of layers. And after integrating the A₁₆ peak, it can be found that the integral value is
linearly related to the layer number of SiP, as shown in Fig. S4 in the ESM. It needs to be pointed out that there is a bad point in the place where the layer number is 11 (the red point), which may be interfered by environment. This finding provides an idea for using Raman spectroscopy as a non-contact measurement of the thickness of nanosheets.

### 3.2 Thickness dependence of the lateral force of SiP nanosheets

The friction between the AFM tip and SiP nanosheets with different layers (1, 2, 5, and 7 layers) under applied loads from 0 to 30 nN is shown in Fig. 3(a). Each set of experimental data was collected from different areas of the SiP sample with the same thickness. Each experimental result was obtained by taking the average friction value of five friction curves to eliminate the experimental error. The error bars indicate the fluctuations of the results. The friction between the AFM tip and SiP nanosheets increases linearly with the applied load and gradually decreases with the increase in layer thickness.

The friction loop is derived for subtraction to obtain the quantitative friction force. Figures 3(b) and 3(c) show the friction loops on the SiP nanosheets at the area of 3 μm × 3 μm and 40 nm × 40 nm, respectively. These friction data are collected when the tip reciprocates on the same line in the selected friction area. The black curve is the trace line, and the red curve is the retrace line. The two lines are theoretically symmetrical about the line with zero friction because the tip moves in opposite directions. However, the two lines are incompletely symmetrical to zero due to the deviation of the experimental instrument. The friction force is obtained by multiplying half of the difference between the two lines in the stable stage, as shown in the light blue area of Fig. 3(c), by using the calibrated lateral force conversion coefficient. The frictional force changes correspondingly with the change in layer thickness in the 3 μm × 3 μm scan size.

The frictional force of the probe moving on the Si sheet and SiP nanosheets with different layers under the load of 2.9 nN is presented in Fig. 4(a). The friction force on the SiP single layer is smaller than that on a silicon wafer. The friction force decreases with the increase in the number of SiP layers and saturates at approximately five layers. The coefficient of friction of multilayer SiP is 0.018, which is smaller than the friction coefficient of BP measured in other experiments [29].

The above experimental results can be explained on the basis of thickness-dependent puckering effect, as shown in Figs. 4(b) and 4(c) [30–32]. When the tip...
slides on a single-layer or two-layer SiP sample, the out-of-plane bending stiffness of the sample is small. Thus, the wrinkles are prone to occur. Large wrinkles increase the contact area between the tip and the sample. More kinetic energy of the tip is converted into the deformation energy of the crystal lattice, thereby increasing the energy dissipation and friction. By contrast, the out-of-plane bending stiffness of the sample is larger for more inner layer interaction with the tip through Van der Waals force. This process is performed when the tip slides on a thick layer (above five layers) or bulk SiP. Consequently, the generation of wrinkles is suppressed, and the lateral force is smaller.

3.3 Velocity dependence of the lateral force of SiP nanosheets

In the macroscopic law of friction, friction is considered to be independent with sliding velocity. However, this law is invalid when the scale is reduced to the microscopic level. For instance, soft materials exhibit a nonmonotonic dependence on speed [33]. Bouhacina et al. [34] found that the silane molecules and the polymer grafted on the coupling agent show a linear increase in friction force with the logarithm of sliding velocity. The speed dependence of SiP nano friction is unclear. Hence, the dependence of SiP nano friction on sliding velocity was investigated.

Figures 5(a) and 5(b) show the average friction between the AFM tip and two-layer SiP nanosheets in the stable stage at low speed (nano scale) and high speed (micron scale), respectively. In this experiment, the low-speed case is 10–640 nm/s, and the high-speed case is 15–160 μm/s. The friction increases exponentially with the increase in sliding speed at the low-speed case. However, the friction force of SiP nanosheets tends to be stable above 40 μm/s at high-speed case.

Enrico et al. [35] thought that the lower scanning speed, the higher the probability of an early thermally activated jump. Therefore, the lateral force increases with the increase in scanning speed. The dependency between friction force $F_l$ and velocity $v$ can be expressed as
where $F_l^0$ is the lateral force needed to induce a jump at $T = 0$, $k_B$ is Boltzmann constant, $T$ is temperature, $a$ is the lattice constant, and $\lambda$ is a length parameter related to the distance involved in slip. The first item in brackets is the ratio of the frequency of stick-slip $v/a$ and a characteristic frequency $f_0$. The second item is the ratio of energy barrier $\lambda k_B a$ ($k_B$ is the effective lateral spring constant) and thermal energy $k_B T$. This relationship, which is dominated by atomic stick-slip, is only suitable for low-speed conditions. With the increase in scanning speed, the factor that dominates the friction during the sliding process gradually becomes the formation of liquid bridge between the tip and the sample. At this time, the higher the speed, the shorter the time of the liquid bridge to form. Thus, the friction gradually decreases. At the micrometer scale, the factors that dominate the friction in the sliding process are between the two factors. Therefore, the influence of the two factors on the friction force is mostly the same when the speed is between 40 and 160 μm/s. Thus, the nanofriction lateral force remains unchanged.

Figures 5(c) and 5(d) show the average friction between the AFM tip and 10-layer SiP nanosheets in the stable stage at low speed and high speed, respectively. The lateral force of the 10-layer SiP increases with the increase in speed at the low-speed case and reaches saturation at the high-speed case. The lateral force of the former is greater than that of the latter, which is consistent with our previous research results. It can be seen more intuitively in Fig. S5 in the ESM that the lateral force of two and ten layers both increases linearly with the logarithm of the sliding speed at nm/s scale.

In the previous friction loop, the curve of friction fluctuates up and down and is called the stick-slip effect. One-dimensional Prandtl–Tomlinson model can be used to explain this phenomenon. In the nanoscale friction situation, the tip moves on the potential energy surface of the sample surface. The atom in front of the tip gradually climbs from a potential well before leaving. This is called the stick process. Then this atom slides away from the potential barrier and slides to the next potential well, which is called the slip process. In a stick-slip cycle, the change in friction is called stick-slip friction. This stick-slip characterizes the frictional vibration state of the system. In the stable friction stage, we can characterize...
these data on the basis of the average value of the deviation between the friction force data and the mean friction force.

In the nanotribology experiment, the scanning speed is usually changed by altering the scanning frequency with a fixed scanning range. However, we found that the frequency and speed have different effects on stick-slip friction (friction vibration) in the experiment. To study the scanning speed effects on friction vibration, we change the scanning speed by changing the scanning distance under the same scanning frequency (1 Hz) first.

The results of frictional vibration analysis at scanning speeds of 10, 20, 40, and 80 nm/s at the same frequency (1 Hz) in stable stage are summarized in Fig. 6(a). The friction vibration was quantified and characterized by using the average value of the absolute difference between the data points and the mean friction force on each friction curve. The statistical results of friction vibration are shown in Fig. 6(b). As can be intuitively seen that minimal difference is observed in frictional vibration at different speeds.

To study the influence of frequency effects on friction vibration, we keep the same scanning speed (80 nm/s) by matching different scanning frequencies and scanning distances. Figure 6(c) shows the frictional vibration at scanning frequencies of 1, 2, 4, and 8 Hz with the same speed (80 nm/s). Figure 6(d) shows the statistical results of friction vibration situations at different frequencies, indicating that the amplitude of frictional vibration increases remarkably with the increase in frequency.

The contact quality evolution model can be used to explain the above phenomenon [40, 41]. The contact quality of the tip with the sample surface increases with time during the sliding process. This condition indicates that when the contact time of the local area becomes longer, the proportion of atoms with high pinning force in the contact area between the tip and the sample increases. Therefore, the contact time in the local area decreases with the increase in scanning speed under the same frequency, resulting in lower contact quality. This condition reduces the resistance barrier that needs to be overcome when the tip moves from the current contact area to the next area, resulting in a slight reduction in frictional vibration. However, the increase in speed results in the vibration of the system, causing a slight increase in frictional vibration.
vibration. The two factors hinder the friction vibration to change with the change in speed at the same frequency, as shown in Fig. 6(b).

The high scanning frequency increases the number of starts and stops when the tip slides over the different distance at the same speed. This condition makes the system change frequently between steady state and unsteady state, resulting in increased vibration of the tip in the vertical direction. As a result, the frictional vibration increases. The fluctuation of the lateral force caused by the beating of the tip in vertical direction is greater than the friction vibration caused by the evolution of the contact quality. Thus, the influence of scanning frequency on the friction vibration is greater than the influence of speed. Therefore, the experimental method of adjusting the scanning speed by controlling the frequency to match the corresponding scanning distance reduces the overall friction fluctuation.

Friction strengthening effects were found in SiP nanosheets. Figure 7(a) shows the lateral force of trace line on 10-layer SiP nanosheets versus scan distance in the range of 3 nm at different velocities of 3, 6, 12, 24, and 48 nm/s. The lateral force curve of nanoscale friction can be divided into two sections. The first section is a process in which the lateral force gradually increases with the scan distance, which is called the friction strengthening effect [40, 42]. The slope of the increasing part represents the degree of friction strengthening. The second section is that the friction reaches saturation at a certain point. The distance required to reach saturation friction is called the friction strengthening saturation distance. Figure 7(b) shows that the friction strengthening saturation distance gradually increases with the increase in speed. This condition shows that the contact friction force between the tip and the sample needs to slide a longer distance to reach saturation under higher speed.

The peak friction force values during the friction strengthening stage under different speeds are extracted to intuitively characterize the degree of friction strengthening. The degree of friction strengthening effect is represented by the slope that is obtained through the linear fitting of peak friction force values, as shown in Fig. 7(c). In the previous study, the friction strengthening effect of few layers of graphene was studied at different speeds, where graphene can dynamically adjust its configuration to

![Fig. 7](a) Lateral force of trace line versus scan distance in the range of 3 nm at different velocities of 3, 6, 12, 24, and 48 nm/s. (b) Relationship between the friction strengthening saturation distance and velocity. (c) Peak force of friction strengthening stage in trace line extracted from (a). (d) Degree of the friction strengthening effect obtained by linear fitting the slope of (c), and the increased friction force at each speed calculated by multiplying the friction strengthening saturation distance and the degree of friction strengthening effect.
change the contact state in the tip-graphene interface [27]. The phenomenon that the degree of friction strengthening decreases with the increase in speed can be explained by the time-dependent contact quality evolution. However, thick 2D materials with high out-of-plane deformation stiffness cannot adjust themselves to change the interface contact state in real time. Thus, the difference in contact quality evolution at different speeds can be ignored. For 10-layer SiP, the number of atoms in the contact interface with the AFM tip is extremely small. This condition makes the degree of friction strengthening at different speeds mostly the same for thick SiP, as shown in the orange bar of Fig. 7(d). The lateral force of the trace curve is calculated by multiplying the saturation distance and the degree of friction strengthening at different speeds, as shown in the blue bar of Fig. 7(d). The calculated lateral force of the trace curve increases with the increase in speed. This condition is consistent with the previous conclusions that the average friction force increases with the increase in scanning speed (nm/s scale).

4 Conclusions

In summary, the tribological properties of SiP nanosheets can be affected by thickness and change with applied load and sliding velocity. The lateral force of SiP nanosheets decreases with the increase in layer thickness and reaches saturation after five layers due to the puckering effect. The coefficient of friction of multilayer SiP is 0.018. The mean friction force increases with the logarithm of the speed first at nm/s scale and then reaches a plateau above 40 μm/s. This condition can be attributed to the phenomenon that the dominant factor of nanofriction gradually translates from atomic stick-slip to the formation of liquid bridges. The influence of frequency on frictional vibration is greater than the speed because frequency has a more significant influence on the change in contact quality. The degree of friction strengthening effect at different speeds is mostly the same (about 0.75 nN/nm), and the friction strengthening saturation distance increases with the increase in speed for thick SiP nanosheets. These results can provide an in-depth understanding of the basic friction properties of SiP, and are helpful for the tribological design of SiP in solid lubrication and microelectromechanical systems.

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