A novel and facile method for detecting the lattice orientation of MoS$_2$ tribological surface using the SPSA process

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HIGHLIGHTS

• Periodic and symmetrical characteristic of the friction force on MoS$_2$ tribological surface were observed
• Distinct frequency spectrum characteristics of the friction signals for various lattice orientations were observed
• Relationship between the frequency and lattice orientation was established
• Single-line-scan power spectrum analysis process for lattice orientation detection was proposed

GRAPHICAL ABSTRACT

ABSTRACT

Lattice orientation detection techniques are crucial for two-dimensional materials as many unusual properties, such as electronic, optical, catalytic and magnetic properties, are closely related to particular lattice orientations. Herein, we propose a novel, low-cost and convenient detection technique, referred to as a single-line-scan power spectrum analysis (SPSA), which is established based on the power spectrum analysis of friction information that is extracted from an atomic-resolution lateral friction microscopy image. By analysing the characteristics of the friction information and the corresponding frequency spectrum obtained from the tribological surface of MoS$_2$, we innovatively establish a relationship between the frequency characteristics and lattice orientation and ultimately propose our SPSA, which enables detection of the direct lattice orientation using the frequency characteristic values of an arbitrary line on an atomic image. The experimental results further verify the effectiveness and convenience of the SPSA process.

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1. Introduction

The direction of a crystal, termed the lattice orientation, is one of the most essential concepts for understanding the crystal structure, and it holds primary significance as a plethora of properties vary with the lattice orientation. In the nanoworld, in which many unique characteristics that span several domains become relevant, such as unusual magnetic behaviour [1], ultrahigh conductivity [2], and novel mechanical properties [3], the impact of the lattice orientation has been investigated intensively for quasizero-, one-, and two-dimensional nanomaterials. For example, the zigzag edges of graphene quantum dots have attracted more interest for experimental investigation than armchair edges due to the presence of localized edge states, which are vital to spintronic...
applications [4]. The mechanical properties and electronic conductivity of carbon nanotubes sensitively rely on their chirality [5], yielding variations in the stiffness, bending rigidity [6], metallic status, and tube-tube contact resistance [7]. For recently emerging two-dimensional materials, many properties related to the lattice orientation have also been revealed. For instance, distinct electronic and optical properties of armchair, diagonal and zigzag structures have been theoretically predicted using density functional theory (DFT) for phosphorene nanoribbons [8]. Recent research has shown that piezoelectric charges prefer to accumulate on the zigzag structures rather than the armchair edges of molybdenum disulfide (MoS2) [9], probably due to the metallic behaviour of the zigzag edges. In brief, research on the lattice orientation of nanomaterials is a subject of unequivocal importance.

Among the vast array of available nanomaterials, real two-dimensional nanomaterials have rarely been investigated due to the absence of experimental evidence indicating their existence. Not until the unexpected success of isolating atomically thin graphene has the existence of a stable two-dimensional crystal been definitively proven [10]. Inspired by this recent achievement, a burgeoning research field has arisen to study the underlying physics of the whole two-dimensional materials family, and this field enables a promising future for the potential applications of two-dimensional materials benefiting from their distinctive features [11, 12]. Nevertheless, graphene has been recently eclipsed by other emerging two-dimensional materials due to their gapless nature [13]. To bypass this hurdle, an increasing number of researchers have been struggling to find other promising alternatives in the two-dimensional family, such as transition metal dichalcogenides (TMDs). MoS2, a representative TMD, possesses multiple functionalities with great potential applications, such as in photonics, valleytronics, nanoelectronics and optoelectronics, rendering it an ideal platform for investigating the fundamental theories underpinning these diverse applications [14, 15]. Digging deeper into the intrinsic physics of MoS2, many novel properties that directly depend on the lattice orientation have been discovered. For example, strong second-order nonlinear optical behaviour has been observed on the boundaries and edges of MoS2 [16]. Anisotropic thermoelctric behaviour has been predicted for MoS2 based on calculation results [17]. Moreover, the piezoelectric response of odd-layered MoS2 was detected on zigzag structures rather than armchair edges [18]. For these reasons, we chose MoS2 as a breakthrough material for lattice orientation research.

To gain insights into the properties related to the lattice orientation, versatile avenues have been pursued. To date, the most common techniques used for detecting the lattice of a material have been transmission electron microscopy (TEM) [19] and scanning tunneling microscopy (STM) [20]. Polarization-dependent Raman spectroscopy has also employed to identify the edges of graphene [21] or the crystallographic orientations of MoS2 based on the polarization dependence of the vibration mode [22]. Additionally, the optical second-harmonic generation (SHG) technique has been utilized to probe the symmetry properties and orientations that originate from the nonlinear polarization that arises from the interactions with a fundamental electric field [23]. Despite the extensive research that has been devoted to lattice detection, there is still significant room for improving the current techniques. The well-known shortcomings of the conventional detection methods, TEM and STM, include the relatively expensive equipment and the time-consuming and tedious pretreatment processes required for the samples, requiring either a special grid or conductive substrate. Using optical techniques, however, clear and atomic images of the samples cannot be obtained. Additionally, the size of a prepared sample is usually limited to a few layers due to the weaker optical response of thicker samples. In view of these imperfect features of the current detection techniques, a question remains: is it possible to develop a convenient and effective lattice orientation detection method?

To resolve this question, in this work, we conducted research on the tribological surface of MoS2 using lateral force microscopy (LFM), which can provide the most direct and comprehensive information that is relevant to the lattice orientation. We initially investigated the periodic and symmetrical variations in the friction force and frequency spectrum characteristics for the various lattice orientations both theoretically and experimentally. Then, by analysing the frequency variation trends within one period, we innovatively established the relationship between the frequency characteristic and the lattice orientation, which enables the direct derivation of the corresponding lattice orientation from the frequency product. Based on the combination of the relationship and the periodic and symmetrical variations, we ultimately proposed the SPSA process. With this method, a random scan line that contains the necessary friction information for FFT can be used to deduce the lattice orientation. Moreover, unlike the aforementioned techniques, high-resolution atomic imaging that usually requires time-consuming and rigorous conditions can be avoided, which greatly facilitates quickly determining the lattice orientation. We also carried out a series of experiments to verify the validity of the SPSA, and the results demonstrate the effectiveness and convenience of the SPSA process. Hence, the SPSA process can be employed for the lattice detection of the tribological surface of MoS2 and has the potential to be broadly applied to other two-dimensional materials.

2. Experimental section

To ensure the accuracy of the experiment, two types of samples, obtained either by mechanical exfoliation (sample-1) or chemical vapor deposition (CVD) (sample-2), were prepared on Si/SiO2 substrates. The bulk crystals of MoS2 were purchased from XFNANO, Nanjing, China, and the CVD-grown MoS2 nanosheets were synthesized at the Shanghai Institute of Optics and Fine Mechanics. After dilution by 1:3, the original MoS2 solution was sonicated for approximately 12 min in 50 Hz in an ultrasonic oscillator (SKS210 LHC). As a relatively clean and flat sample surface was a necessary prerequisite to guarantee the success of the experiment, atomic force microscopy (AFM) (Icon, Bruker) was first utilized to verify the surface morphology of the two types of sample with areas of approximately 3.1 × 8.2 μm and 23.1 × 11.1 μm (Fig. 1a and d, respectively). The sizes and heights the two samples were also measured, showing that the thicknesses of the two samples were 2.9 nm and 0.7 nm, respectively (Fig. 1b and e). In addition to AFM, Raman scattering spectra, which have been proven to be a noncontact, nondestructive and powerful tool to observe the number of sheets of a sample, were measured as a crucial supplement. A 532-nm excitation laser was focused on the nanosheets, and two distinct peaks appeared for the in-plane E2g and out-of-plane A1g modes of the corresponding samples. As shown in Fig. 1(c) and 1(f), the distances between the E2g mode and A1g mode are 24.3 and 18.1 cm−1, which agree well with the previous results in the literature [24], indicating that the two samples are four-layer and monolayer nanosheets (For sample-1, Raman spectroscopy was performed on the regions with a lower height.)

After the thicknesses of the samples were confirmed, lateral force microscopy (LFM) was subsequently performed on the relatively flat regions of the sample surfaces to obtain atomic images and atomic-scale friction information. The friction measurements of MoS2 were performed using the contact mode of a multimode scanning probe microscope (SPM) in air under ambient conditions (43 to 47% relative humidity, atmospheric pressure and 20–24 °C room temperature (293 K to 297 K); the slight temperature variation did not affect the LFM measurements). The scan size and rate were 10 × 10 nm and 29.5 Hz, respectively, using a normal contact silicon nitride probe with a 0.02 N/m spring constant. To ensure high-quality imaging, the scan direction of the cantilever was always maintained at 90°, and the positions of the as-prepared samples remained constant during the LFM measurements. In addition to the above conditions, an ultralow noise measurement environment was required to obtain high-quality atomic images. Thus, a floating platform and other noise enclosure equipment
made of metal and absorbent cotton materials were employed to isolate the measurement system from external disturbances.

3. Results and discussion

3.1. Theoretical analysis and simulation of the friction signals

Fig. 2(a) shows a schematic image of the LFM system. According to a previous report and early research on the Tomlinson model [25], the LFM system can be simplified into several components (Fig. 2b). When LFM experiments are performed on a sample, the probe will continuously jump along the surface probe. Ideally, the parameters of the motion, such as the elasticity of the cantilever, spring constants, effective mass, and damping term, can be modelled. The loading force is also regarded as a constant. Based on this approximation, a stable equilibrium state between the probe and sample surface will be reached, and the motion can be described as follows

\[ m_x \ddot{x}_t = k_x(x_M - x_t) - \frac{\partial V(x, y)}{\partial x} - \gamma_x \dot{x}_t, \]

\[ m_y \ddot{y}_t = k_y(y_M - y_t) - \frac{\partial V(x, y)}{\partial y} - \gamma_y \dot{y}_t, \]

where \( m_x, m_y, k_x, k_y, \) and \( \gamma_x, \gamma_y \) are the effective mass, elasticity of the cantilever, and damping terms respectively, and \( x_t, y_t \) and \( x_M, y_M \) denote the positions of the probe and the microscope support, respectively.

To solve the equation above, a simple and appropriate probe-sample interaction should be utilized. Based on the theoretical and experimental analysis provided in previous research [26], the original interaction

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**Fig. 1.** AFM imaging and Raman spectra of the mechanically exfoliated and CVD-grown MoS2 nanosheets. (a) and (d) are the AFM images for sample 1 and sample 2. The white squares on (a) and (d) indicate the relatively flat regions that were confirmed using AFM. (b) and (e) show the height profiles for the X-X’ and Y-Y’ positions on sample 1 and sample 2. (c) and (f) are the Raman spectra for the different regions on the two samples.

**Fig. 2.** Experimental apparatus and equivalent model. (a) Schematic illustration of the LFM equipment. (b) Equivalent motion model of the LFM and cross-sectional view of the MoS2.
relationship should consider the moving trajectories of the various lattice orientations, and it can be modified as follows

\[ V(x_i, y_i) = V_0 \cos \left( \frac{2\pi}{R'} x_i \right) \cos \left( \frac{2\pi}{R'} y_i \right) \]  

(3)

\[ R' = R \cos \theta \]  

(4)

where \( V_0 \) is the barrier potential constant with an estimated value of 1.0 ev, \( a_y \) and \( a_y \) are the lattice constants of MoS\(_2\), \( \theta \) denotes the lattice orientation, and \( R \) is the length of the probe path. Notably, there is no need to calculate the specific values of \( R \) for the various lattice orientations, which can be offset in the subsequent analysis for the frequency spectrum.

Considering the sandwich-like construction of MoS\(_2\), the interaction between the probe and MoS\(_2\) should consist of two parts: One part is the interaction between the probe and the upper Mo layers, and the other is the probe interaction with the lower Mo layers. As the interaction between the Mo layers is much weaker than that between the S layers, for the purpose of simplicity, the sandwich structure of MoS\(_2\) can be simplified as the form shown in Fig. 3(b).

Based on the analysis and parameters above, the friction signals for various lattice orientations within 360° were simulated (the simulation was carried using 10° increments) using Matlab software (MathWorks, USA, version 7.0). The simulation results can be found in Fig. S1 of the Supporting Information, which clearly exhibits a 180-degree period wave shape and wavenumbers. Therefore, we calculated the atomic-scale friction force of the various lattice orientations for the two samples. The lateral force was obtained using the following equation by substituting the parameter values [27]:

\[ F_l = \frac{Gw^2}{3(h + t/2)} S_l V_l \]  

(5)

where \( h \) describes the height of the probe and \( G, w, l, t \) are the shear modulus, width, length and thickness of the cantilever, respectively. The values of the probe parameters can be found in Fig. S4(a). Moreover, \( V_l \) is the lateral voltage output, and its value at three locations of the various lattice orientations can be seen in Fig. 5(a). The average values of \( V_l \), or the fit curves, of both sample 1 and sample 2 exhibited a 90-degree symmetrical trend, and \( S_l \) is the lateral sensitivity of the photodetector, which can be regarded as the normal sensitivity by postulating that the photodetector rotated symmetrically. This variable was thereby estimated using

\[ S_l = S_n = \frac{3 \, dx_n}{2 \, dV_n} \]  

(6)

where \( \frac{dx_n}{dV_n} \) denotes the slope of the force curve shown in Fig. S5(b), and the calculated value is 138.63 nm V\(^{-1}\). Thus, according to the relevant parameters of the probe and the slope curve in Fig. S4, the friction forces for various angles can be expressed as \( F_v \approx 0.049nN/V \). Under the friction forces, the loading force could also be calculated using \( F_z = kS_n V \), based on the above parameters, where \( k \) is the normal-spring constant from the measured thermal noise and \( V \) is the corresponding voltage output of the photodetector. In our experiment, the horizontal deflection was set as \(-1 V\) with a 0 V setpoint, and the normal-spring constant is 0.02 N/m. Thus, the value of the loading force is approximately 2.77 nN.

As displayed in Fig. 5(b), we made a comparison between the real and simulated friction forces (the simulated friction force was obtained by simply averaging the absolute values in Fig. S1). From the

![Fig. 3. Structure and lattice constants of the MoS\(_2\). (a) Top view of the MoS\(_2\). The grey and blue spheres indicate the S and Mo atoms of MoS\(_2\), respectively. (b) Top view of the MoS\(_2\), where \( w_0 \) and \( w_1 \) indicate the frequency components. The red and black dotted lines are perpendicular auxiliary lines that we added for the calculation of \( w_0 \) and \( w_1 \). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)](image-url)
comparison, both the simulated and experimental results exhibited an anisotropic friction force distribution with a 180-degree period variation and 90-degree symmetry characteristic. More interestingly, the zigzag lattice orientations, such as 0°, 60°, 120°, and 180°, apparently possessed a larger friction force than the other lattice orientations. Although a great discrepancy existed between the theoretical and experimental results, probably because of the unavoidable experimental error and the fact that the mechanism underlying this anisotropic behaviour remains unclear, the discovery of this phenomenon might help to establish a closed-loop friction force feedback system and thereby realize real-time lattice-oriented operation and fabrication. Additionally, the experiments were carried out under 43 to 47% relative humidity, which would inevitably lead to an increase in the friction force when the water meniscus formed between the probe and MoS2 interface. Thus, based on the experimental data in reference [28], the lateral friction force for the experimental conditions would experience an approximately 1.5-2-fold increase compared with values for the dry environment. Within the 43 to 47% relative humidity range, the increase in the amplitude of the lateral friction force was estimated to less than 10%, which did not affect the overall variation trend of the friction force.

3.3. Frequency spectrum characteristics of the friction signals

Based on the periodic variations, a further analysis of the friction signals of the MoS2 nanosheets was simplified within one period. However, comparing the directions of the friction signals from 0 to 180° was quite difficult due to the huge number of friction signals as the friction information was obtained from three different locations for each lattice orientation. What is worse, the friction force information contained too much uncertainty and was easily affected by external experimental factors. As seen in Fig. 5 (a), the friction forces of the different locations, L-1, L-2, and L-3, varied significantly, even for the same lattice orientation. Therefore, an exclusive characteristic or a definite relationship between the friction signals and the lattice orientation should be established.

To further simplify the friction signals within one period, fast Fourier transform, FFT, a powerful algorithm with great efficiency, was employed for the various lattice orientations, which allowed for rapid signal processing and, more importantly, greatly eliminated the undesirable disturbances of the signals that may have originated from the equipment and environmental noise. The frequency spectra of the
various lattice orientations for sample 1 and sample 2 are shown in Fig. S5. After analysing the characteristics, we found that the frequency spectra for the various lattice orientations could be divided into four categories according to the number of frequency peaks, one peak, two peaks, three peaks and three or four peaks, which are clearly shown in Fig. 6.

To explore this scenario, the modified interaction potential was once again analysed. After converting the angular frequency, the natural frequency components that the interaction potential contained were written as

\[ w_1 = \left| \left( \cos \theta + \sin \theta / \sqrt{3} \right) / R \right| \]

\[ w_2 = \left| \left( \cos \theta - \sin \theta / \sqrt{3} \right) / R \right| \]

Thus, for the special angles, such as the zigzag and armchair lattice orientations, the values of the frequency were obtained by substituting the corresponding angle. Then, for the zigzag lattice orientation, \( w_1 = \frac{1}{2} = \frac{1}{\sqrt{3}} \) and for the armchair lattice orientation, \( w_1 = \frac{1}{\sqrt{3}} \) and \( w_2 = \frac{1}{\sqrt{3}} \), which were used to explain the one- or two-frequency peaks for these two cases.

However, for the general lattice orientation, the probe motion on the MoS₂ surface was irregular and may have generated another two frequency components, \( w_b \) and \( w_c \), besides the two natural frequency components, as shown in Fig. 3(b). According to the triangle cosine theorem and the lattice constants of the S atom layer, \( w_b \) and \( w_c \) were expressed as

\[ w_b = \left| R \times \cos \left( 60^\circ - \theta \right) \right| \]

\[ w_c = \left| R \times \cos \left( 60^\circ + \theta \right) \right| \]

By combining the natural frequency components \( w_1 \) and \( w_2 \) with the motion frequency components \( w_b \) and \( w_c \), the frequency distributions for the various lattice orientations were obtained. As depicted in Fig. 7(a), the frequency distribution also exhibited the same periodic and symmetric features as those of the friction force. In Fig. 7(c), the characteristics of the frequency distribution within one period were

![Image](image-url)
Further analysed, showing that the values of the frequency within a certain angle range, 20° to 40°, were very close. For the angle range of 40° to 80°, the values for frequency component \( w_2 \) were much lower than the others. As the LFM experiment was carried out under ambient conditions, the frequency resolution was easily affected by many factors, such as the equipment noise, experimental error, and temperature drift. Thus, the minor frequency differences or tiny frequency values may not have been detected, and the general lattice orientation (Fig. 6c and d), could exhibit three or four frequency peaks. Overall, the experimentally observed frequency spectrum variations of the various lattice orientations could be theoretically explained using a combination of the natural and motion frequency components.

By comparing the formations of the frequency components, we found that both the natural and motion frequencies consisted of two parts: the angle-related expression and the probe motion path \( (R') \). The quotient or product of these two parts constituted the final expression for the natural and motion frequency components. Thus, by multiplying these two types of frequency components, the probe motion path \( (R') \) could be offset, and the angle-related expression was maintained in the frequency product. In this way, the complex calculation of the probe motion path for the various lattice orientations was avoided, and a relationship between the frequency and the lattice orientation could be established. The four cases for the frequency product distribution within 180° are shown in Fig. 7(b). Due to the 90-degree symmetrical characteristic of the frequency component, the products of the four cases exhibited 45-degree symmetry and a 90-degree periodic variation.

To select the appropriate frequency products from the four cases, the real frequency products for various the lattice orientations were calculated. Based on the frequency spectra in S5, the two strongest frequency peaks were chosen, and their products are summarized in Table 1 (Supporting Information). The theoretical and experimental frequency products were chosen, and their products are summarized in Table 1 (Supporting Information). The theoretical and experimental frequency products within 90° are displayed in Fig. 7(d). Clearly, the frequency product \( w_1 \times w_2 \) best fits the experimental results. Thus, the frequency product \( \delta \) was expressed as

\[
\delta = w_1 \times w_2 = \left| \cos \theta \frac{\sin 2\theta}{\sqrt{3}} / R' \right| \times \left| R' \cos(60° - \theta) \right|
\]

\[
= \cos \theta + \sin \theta / \sqrt{3} \times \cos(60° - \theta)
\]

\[
= \frac{1}{2} + \frac{1}{\sqrt{3}} \sin \theta
\]

\[
= \frac{1}{2} + \frac{1}{\sqrt{3}} \sin \theta
\]

(11)

Given the frequency product, \( \delta \), the lattice orientation, \( \theta \), was calculated using the relationship above.

3.4. SPSA process and experiment verification

Based on the relationship between the frequency product, \( \delta \), and lattice orientation, \( \theta \), a lattice detection process, SPSA, was developed. First, the atomic image of the sample surface was acquired by conducting an LFM experiment. Then, the friction signals were obtained from a random lattice orientation and transformed using FFT. The two strongest frequency peaks were then extracted and multiplied. The product, \( \delta \), was substituted into the following deformation of Eq. (11).

\[
\sin 2\theta = \sqrt{3} \left( \delta - 1 - \xi \right) \left( 1 - \frac{1}{2} \right)
\]

(12)

where \( \xi \) is the estimated error, with a value of approximately 3%–5%. The calculated values of \( \delta \) were compared with the theoretical values, and the closest value was chosen. Due to the 45-degree symmetry of the frequency spectrum, the ultimate value of \( \theta \) were further confirmed. As the special angles, such as the zigzag and armchair lattice orientations, had fewer frequency peaks (one or two), their frequency spectra were more stable and reliable than those of the general lattice orientation and could serve as proof of the lattice detection results. By rotating the line anticlockwise by an angle of \( \theta \), the friction signal for the new location was acquired. After the FFT, if the characteristic of the frequency spectrum was consistent with that of the zigzag lattice orientation (0°), then the lattice orientation was confirmed as \( \theta \). Otherwise, the line continued to be rotated anticlockwise by angles of 90°, 90° + \( \theta \), and 180°, respectively, and a judgement was made using the same method. Fig. 8 shows a schematic illustration of the SPSA process.

The most prominent part of the SPSA process is the time-saving atomic-imaging step. As the redundant information was effectively eliminated using the FFT, frequency peak extraction and error-involved calculation, the useful frequency information related to the lattice orientation was retained to the greatest extent. More specifically, as long as the friction signal for a certain lattice orientation was acquired from the atomic image, the angle-related information it contained could be extracted using the subsequent process. Hence, the original atomic image was not necessarily complete and clear, but the low-quality atomic image that could be realized in a short time was also applicable to the entire information extracting method. It is worth noting that the mechanically exfoliated MoS₂ nanosheet or CVD grown MoS₂ nanofilms would inevitably contain structural imperfections, such as point or extended lattice defects, and these defects would greatly influence the electrical and optical properties of MoS₂ [29,30]. However, for our case, the localized imperfect MoS₂ surface did not affect the lattice orientation detection. On one hand, the scan area of the LFM images was only a few square nanometers, and the random line on the LFM image that we used to extract the friction signal for a certain lattice orientation was also only a few nanometers in length, which means that of choosing a random line in the defect region was a low-probability event. Even if this happened, the friction signals that were obtained from the defect region were easily distinguished from the normal ones. As shown in Fig. 5(b), the corresponding power spectrum of the friction signals that occurred within the defect regions obviously did not exhibit frequency peaks. This phenomenon is quite reasonable due to broke lattice structure in the defect regions and the destruction of the interactions between the probe and the atoms. Therefore, the normal frequency characteristics were not observed. In this case, we would randomly choose another line on the LFM image for lattice orientation detection.

To verify the validity of the SPSA process, we carried out several subsequent experiments. After approximately 5–10 min of the LFM operation, a relatively fuzzy atomic image with a low atomic resolution was obtained, as displayed in Fig. 9(a). By analysing a random line, the friction signal of the scanned line was acquired. After calculating the FFT, the frequency peaks were determined to be 0.61 and 1.78, respectively. The corresponding frequency product was 1.08612. According to Eq. (10), with a 3%–5% error, the frequency products were modified as 1.05353, 1.0426752 and 1.031814, respectively, and the calculated lattice orientation was derived as the angle between 34°–36°. Compared with the theoretical frequency products for 34°–36°, 1.03531, 1.04253 and 1.04909, respectively, the error-involved frequency product of 1.0426752, was undoubtedly the closest value. Then, the preliminary result of the detected lattice orientation was 35°. By rotating anticlockwise by 35°, as depicted in Fig. 9(d), the obtained friction signal is shown in Fig. 9(e). After applying an FFT, only one peak was observed from the frequency spectrum, which is consistent with the zigzag lattice orientation (0°). In Fig. 9(g)–(i), after rotating anticlockwise by 5°, the frequency characteristic of the armchair lattice orientation (30°) was observed, which also confirmed that the lattice orientation of the scan line was 35°. The SPSA-based lattice orientation for the other lines of this atomic image and the comparison between the fuzzy and clear atomic images can be seen in Fig. 57 and 58, which further affirms the reliability and practicability of the proposed SPSA process.
Fig. 8. Schematic illustration of the SPSA process. (a) through (f) are the operating steps.

Fig. 9. Experimental verification of the SPSA process. (a) Relatively low-resolution atomic image and analysis of a random line on the image. (b) and (c) are the friction signals of the white line in (a) and the corresponding frequency spectrum. (d) and (g) provide further evidence of the lattice orientation after anticlockwise rotation by 35 and 5°. The dotted lines are obtained by rotating the solid lines by 35 or 5°. (e), (f) and (h), (i) are the obtained friction signals and the corresponding frequency spectra for the dotted lines in (d) and (g), respectively.
4. Conclusion

In summary, we developed a simple and low-cost lattice orientation detection technique by analysing the friction information and frequency characteristic of the tribological surface of MoS₂. From the high-quality atomic images obtained using LFM, we experimentally observed the 180-degree periodic variations and 90-degree symmetrical characteristics of the friction force for the first time. With the aid of FFT, the frequency spectra characteristics of the friction force varying within 180-degree increments were investigated and explained using the theoretical analysis of the frequency components of the probe motion. Based on these analyses, we innovatively established a relationship between the frequency products and lattice orientation, and ultimately proposed the SPSA process, which enabled lattice orientation detection by analysing tribological MoS₂ information from a single scan line. Our proposed SPSA process provides a robust alternative to conventional techniques due to its obvious advantage of enabling convenient, time-saving and feasible measurements. Further optimization is underway towards realizing a rapid lattice orientation regime that is applicable for other two-dimensional materials.

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Appendix A. Supplementary data

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