

# Friction characteristics of mechanically exfoliated and CVD-grown single-layer MoS<sub>2</sub>

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**Abstract:** In this work, the friction characteristics of single-layer MoS<sub>2</sub> prepared with chemical vapor deposition (CVD) at three different temperatures were quantitatively investigated and compared to those of single-layer MoS<sub>2</sub> prepared using mechanical exfoliation. The surface and crystalline qualities of the MoS<sub>2</sub> specimens were characterized using an optical microscope, atomic force microscope (AFM), and Raman spectroscopy. The surfaces of the MoS<sub>2</sub> specimens were generally flat and smooth. However, the Raman data showed that the crystalline qualities of CVD-grown single-layer MoS<sub>2</sub> at 800 °C and 850 °C were relatively similar to those of mechanically exfoliated MoS<sub>2</sub> whereas the crystalline quality of the CVD-grown single-layer MoS<sub>2</sub> at 900 °C was lower. The CVD-grown single-layer MoS<sub>2</sub> exhibited higher friction than mechanically exfoliated single-layer MoS<sub>2</sub>, which might be related to the crystalline imperfections in the CVD-grown MoS<sub>2</sub>. In addition, the friction of CVD-grown single-layer MoS<sub>2</sub> increased as the CVD growth temperature increased. In terms of tribological properties, 800 °C was the optimal temperature for the CVD process used in this work. Furthermore, it was observed that the friction at the grain boundary was significantly larger than that at the grain, potentially due to defects at the grain boundary. This result indicates that the temperature used during CVD should be optimized considering the grain size to achieve low friction characteristics. The outcomes of this work will be useful for understanding the intrinsic friction characteristics of single-layer MoS<sub>2</sub> and elucidating the feasibility of single-layer MoS<sub>2</sub> as protective or lubricant layers for micro- and nano-devices.

**Keywords:** atomic force microscope; chemical vapor deposition; grain boundary; friction, mechanical exfoliation; MoS<sub>2</sub>

## 1 Introduction

Atomically thin molybdenum disulfide (MoS<sub>2</sub>) exhibits remarkable thermal [1], electrical [2, 3], and optical [4] properties that differ from those of bulk MoS<sub>2</sub>. Various electronics [5, 6], optoelectronics [7, 8], and sensors [9, 10] based on atomically thin MoS<sub>2</sub> have been proposed due to these material properties. In addition, the superior mechanical properties [11, 12] and low frictional characteristics [13, 14] of atomically thin MoS<sub>2</sub> suggest that it has potential as an effective protective or lubricant layer [15, 16] for micro- and

nano-devices that experience contact sliding during operation.

Recently, the friction characteristics of two-dimensional materials have been extensively investigated using atomic force microscope (AFM). However, most research has focused on graphene [14, 17–21], and the friction characteristics of atomically thin MoS<sub>2</sub> have been reported in only a few studies [14, 22–24]. For example, the friction of atomically thin MoS<sub>2</sub> was found to decrease as the number of layers increased, similar to other two-dimensional materials [14]. This friction behavior was explained based on the puckering

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and electron-phonon coupling effects [14, 17]. Friction characteristics of single-layer MoS<sub>2</sub> were also compared to that of single-layer graphene [22]. In addition, the friction characteristics of single-layer MoS<sub>2</sub> deposited on SiO<sub>2</sub>, mica, and *h*-BN substrates were investigated. The results showed that friction was the lowest for the *h*-BN substrate, due to a significant reduction in surface roughness [23]. In addition, the friction of single-layer MoS<sub>2</sub> was found to slightly increase with increasing humidity [24]. It was further demonstrated that adding atomically thin MoS<sub>2</sub> to a liquid lubricant can enhance the tribological performances [25]. However, to properly implement atomically thin MoS<sub>2</sub> as a protective or a lubricant layer, more data should be gathered over a wide variety of experimental conditions.

Various methods, such as mechanical exfoliation [9, 26], lithium intercalation-assisted exfoliation [24], liquid exfoliation [27], and physical vapor deposition [28] have been used to prepare atomically thin MoS<sub>2</sub>. Thinning using laser-induced ablation [29], thermal annealing [30], and plasma [31] have also been demonstrated for fabricating single-layer MoS<sub>2</sub>. However, the lateral size of MoS<sub>2</sub> layers prepared using these methods is often relatively small. In addition, the deposition of MoS<sub>2</sub> with controlled size and shape onto specific locations using these methods is often challenging due to technical difficulties. Chemical vapor deposition (CVD), based on the vapor phase reaction of Mo and S, is one of the most practical methods for large area synthesis [32–35], and hence can be appropriately used to facilitate atomically thin MoS<sub>2</sub> layers as protective or lubricant layers. However, in contrast to the optical [36], electrical [34, 35], and mechanical properties [37], the friction characteristics of CVD-grown MoS<sub>2</sub> have not yet been explored. Considering that CVD-grown MoS<sub>2</sub> can have large variations in grain size, crystalline orientation, and number of defects [32–35], which in turn affect friction characteristics, a systematic approach is needed to fundamentally understand the intrinsic friction characteristics of CVD-grown MoS<sub>2</sub>.

In this work, the friction characteristics of single-layer MoS<sub>2</sub> prepared using CVD at various temperatures were systematically investigated using AFM. In particular, given that mechanically exfoliated MoS<sub>2</sub>

has high crystallinity with a small number of defects, the friction characteristics of CVD-grown single-layer MoS<sub>2</sub> were compared to those of mechanically exfoliated single-layer MoS<sub>2</sub>. The MoS<sub>2</sub> specimens were also characterized using Raman spectra to understand the crystalline qualities of the specimens. The outcomes of this work are expected to aid in evaluating the feasibility of single-layer MoS<sub>2</sub> as protective or lubricant layers for micro- and nano-devices, and optimize CVD growth conditions from a tribological point of view.

## 2 Experimental details

The MoS<sub>2</sub> specimens were deposited onto a SiO<sub>2</sub>/Si substrate using mechanical exfoliation and CVD. The thickness of the thermally grown SiO<sub>2</sub> layer on the Si wafer was about 300 nm. After mechanical exfoliation of the MoS<sub>2</sub> flakes using natural crystalline MoS<sub>2</sub> (SPI supplies), they were carefully examined using an optical microscope to ensure the specimens were free from tape residue. Then, the locations of the single-layer MoS<sub>2</sub> were identified based on the thickness-dependent optical contrast. For the CVD process, MoO<sub>3</sub> (99%, Aldrich) and sulfur powders (99.5%, Alfa) were used as sources of Mo and S, respectively. Crucibles containing MoO<sub>3</sub> and sulfur were placed in the center of a furnace and in the upstream zone of a quartz tube, respectively. The substrates were placed face down above the crucible containing MoO<sub>3</sub>. Initially, the quartz tube was pumped down to a base pressure of 60 mTorr and purged with high-purity N<sub>2</sub> gas to eliminate oxygen. Then, the temperature was gradually increased to 400 °C at a rate of 25 °C/min and maintained for 30 min, while the pressure was set to ~700 mTorr with a 100 sccm N<sub>2</sub> gas flow. After the pressure was reduced to atmospheric conditions using 10 sccm N<sub>2</sub> gas flow, the temperature for the reaction was increased to 800 °C, 850 °C, and 900 °C at a rate of 25 °C/min and maintained for 5 min for the growth of single-layer MoS<sub>2</sub> before cooling to room temperature.

After preparation of the MoS<sub>2</sub> specimen, Raman measurements were performed using a confocal Raman system (Alpha300R, Witec) to evaluate the crystalline quality of the MoS<sub>2</sub> layer; the excitation laser with a wavelength of 532 nm was used. For CVD-grown

MoS<sub>2</sub>, the isolated flakes were initially formed and then merged into a continuous film during the CVD process. Hence, a laser with a spot size of ~720 nm was carefully focused on isolated MoS<sub>2</sub> flakes to minimize the effects of the grain boundary. In addition, to eliminate laser-induced thermal effects [38, 39] and particle formation [40], the laser power on the specimen surface was limited to below 0.5 mW. The Raman spectra were collected using a 100× objective (NA = 0.9) for 10 s in ambient conditions.

Topographic images of the MoS<sub>2</sub> specimens were obtained using the AFM intermittent contact mode and Si tips with a nominal normal spring constant of 2 N/m (AC240, Olympus). The friction loops were obtained under normal forces ranging from 0 nN to 20 nN using a Si tip with a nominal normal spring constant of 0.2 N/m (PPP-LFM, Nanosensors). For quantitative force measurements using AFM, normal [41] and lateral force [22, 42] calibrations were performed. The force calibration results showed that the normal spring constant and lateral force sensitivity of the Si tip used for friction loop measurements were 0.47 N/m and 5.85 mV/nN, respectively. More than 10 friction loops were obtained at 10 different locations on the specimens under each normal force. In particular, considering that AFM tip wear can readily occur due to contact sliding [43], the friction loops were obtained under increasing and decreasing normal forces to minimize the effects of tip wear on the friction force measurements. In the friction loop measurements, scan distance and sliding speed were set to 300 nm and 375 nm/s, respectively. After the friction loop measurements, friction force images of the grain boundaries of the CVD-grown MoS<sub>2</sub> specimens were obtained to analyze the difference in friction characteristics at the grains and grain boundaries. These friction force images were obtained using the AFM contact mode under 3 nN normal force and 630 nm/s sliding speed.

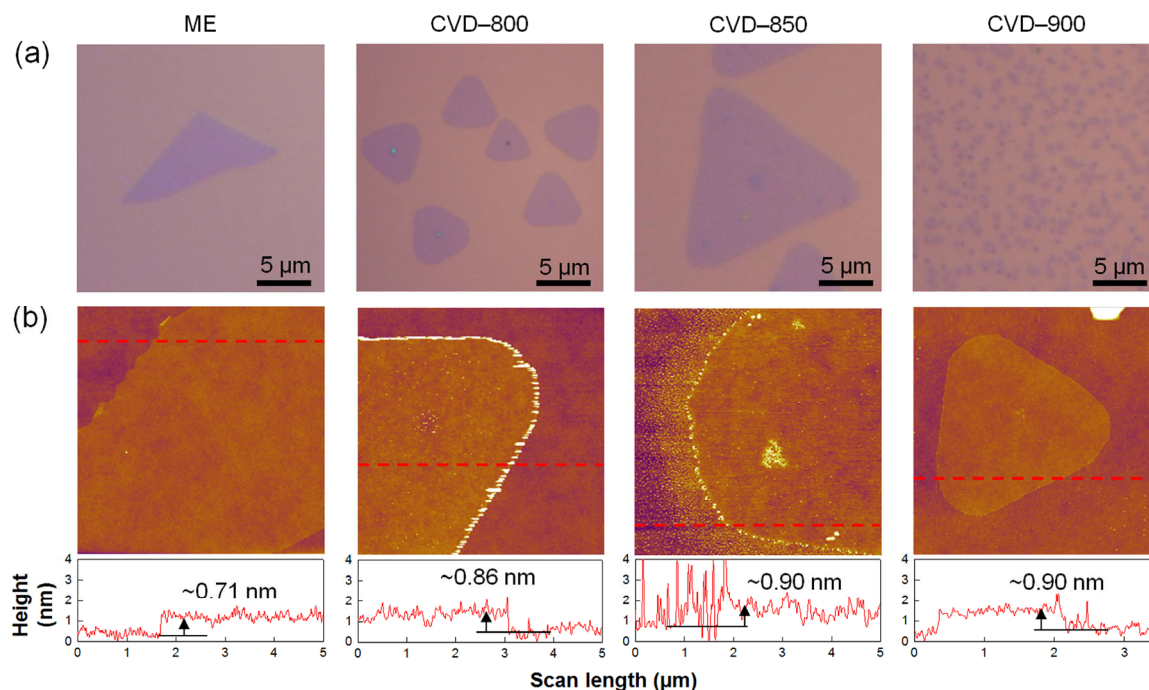
In addition to friction force measurements, the adhesion forces of the specimens were determined using the force-distance curve [44]. The Si tip used for the friction loop measurements was also used for the force-distance curve measurements. Force-distance curves were obtained before and after the friction loop measurements to further monitor AFM tip wear

[45]. More than 10 force-distance curves were obtained at different locations for each specimen. All experiments were performed under ambient conditions (23 °C and 35% relative humidity).

### 3 Results and discussion

Figure 1(a) presents optical microscope images of the mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C on the SiO<sub>2</sub>/Si substrate. For comparison, optical microscope images of isolated flakes of the CVD-grown MoS<sub>2</sub> are shown in Fig. 1. The shape of the mechanically exfoliated MoS<sub>2</sub> was random with a nominal size of about 10 μm, while the CVD-grown MoS<sub>2</sub> exhibited a triangular shape with different sizes depending on growth temperature. The grain sizes of CVD-grown MoS<sub>2</sub> increased from 5 μm to 15 μm when the temperature increased from 800 °C to 850 °C. However, the grain sizes of CVD-grown MoS<sub>2</sub> significantly decreased, to about 3 μm, at 900 °C. This grain size dependence on CVD temperature is often noted in the literature. For example, it was demonstrated that the CVD-grown MoS<sub>2</sub> grain size increases when the temperature increases to about 830 °C, associated with production and diffusion of active mobile species, and then the grain size decreases due to thermal etching when the temperature reaches 900 °C [36]. Figure 1(a) indicates that the grain size of CVD-grown MoS<sub>2</sub> at 850 °C was the largest, which is consistent with the results from previous research [36].

Considering that surface topography can significantly affect the friction characteristics of two-dimensional materials [20, 23], the MoS<sub>2</sub> specimens were examined using AFM prior to the friction loop measurements. The AFM topographic images of mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C obtained with the intermittent contact mode are shown in Fig. 1(b). As shown, the surfaces of the mechanically exfoliated and CVD-grown MoS<sub>2</sub> were generally flat and smooth. However, for the CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C, particles were observed on the substrate surfaces around the flakes. In particular, a large number of particles was observed on the substrate around the CVD-grown MoS<sub>2</sub> flakes at 850 °C. These particles



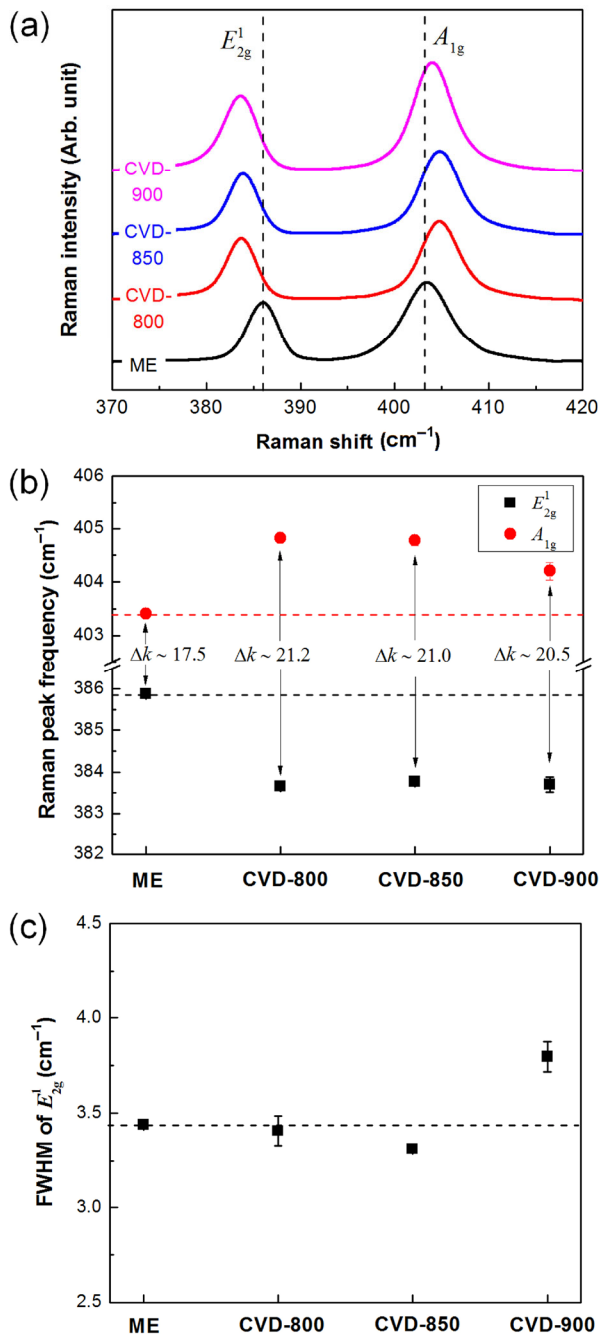
**Fig. 1** (a) Optical microscopy and (b) AFM topographic images of mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C. The red dashed lines indicate the locations of the cross-sectional profiles shown in (b).

could have contained by-products formed during the deposition process. In contrast to the CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C, the substrate surface around the CVD-grown MoS<sub>2</sub> flakes at 900 °C was relatively clean, possibly a result of thermal etching [36]. The surface roughness values of the MoS<sub>2</sub> specimens were determined from AFM topographic images obtained at five different locations within a 500 nm × 500 nm scanning area. The measured surface roughness values of the mechanical exfoliated MoS<sub>2</sub> and CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C were  $0.16 \pm 0.01$  nm,  $0.20 \pm 0.01$  nm,  $0.25 \pm 0.01$  nm, and  $0.16 \pm 0.01$  nm, respectively. These surface roughness values indicate that the MoS<sub>2</sub> specimens used in this work had flat and smooth surfaces.

The heights of the MoS<sub>2</sub> specimens were determined from the cross-sectional height profiles shown in Fig. 1(b). The height of the MoS<sub>2</sub> prepared using mechanical exfoliation was 0.71 nm. However, the heights of the CVD-grown MoS<sub>2</sub> were slightly larger, ranging from 0.86 nm to 0.90 nm. These values were also larger than the theoretical thickness of single-layer MoS<sub>2</sub> (0.62 nm). These discrepancies between the measured and theoretical thicknesses might have been

due to with the presence of adsorbents below the MoS<sub>2</sub> layer, interactions between the MoS<sub>2</sub> layers and substrate [46, 47], and/or AFM measurement uncertainty [48]. Nonetheless, the thicknesses of the single-layer MoS<sub>2</sub> specimens prepared using mechanical exfoliation and CVD were comparable to those of single-layer MoS<sub>2</sub> on a SiO<sub>2</sub>/Si substrate.

To understand the crystalline qualities of the MoS<sub>2</sub> specimens, Raman measurements were performed. Figure 2(a) shows examples of the Raman spectra of mechanically exfoliated MoS<sub>2</sub> and CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C. Two characteristic Raman peaks for MoS<sub>2</sub>,  $E_{2g}^1$ , and  $A_{1g}$ , resulting from the in-plane vibration of Mo-S atoms and out-of-plane vibration of S atoms, respectively [47], are clearly shown in Fig. 2(a). The thickness and crystalline quality of the atomically thin MoS<sub>2</sub> layer can be identified using the frequencies of the  $E_{2g}^1$  and  $A_{1g}$  peaks and the frequency separation between the two peaks,  $\Delta k$  [32, 47, 49]. The frequencies of the  $E_{2g}^1$  and  $A_{1g}$  peaks for the mechanically exfoliated and CVD-grown MoS<sub>2</sub> are summarized in Fig. 2(b), along with  $\Delta k$ . The measured value of  $\Delta k$  for the mechanically exfoliated MoS<sub>2</sub> was  $17.5 \text{ cm}^{-1}$ , which corresponds to single-layer



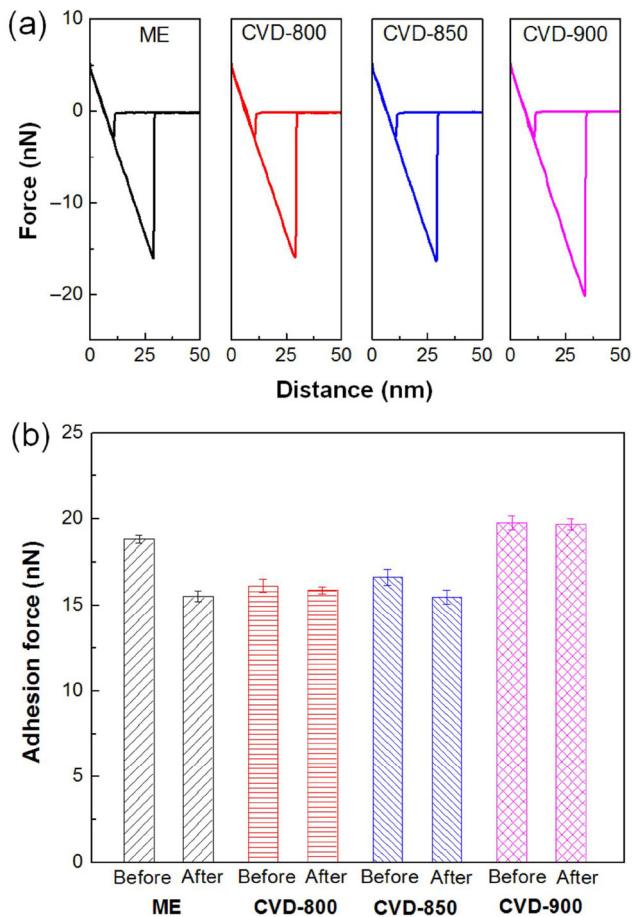
**Fig. 2** (a) Raman spectra, (b) frequencies of the  $E_{2g}^1$  and  $A_{1g}$  peaks and the frequency separation,  $\Delta k$ , and (c) FWHM of the  $E_{2g}^1$  peak of the mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C. In (a), (b), and (c), the frequency of the  $E_{2g}^1$  and  $A_{1g}$  peaks and FWHM of the  $E_{2g}^1$  peak of the mechanically exfoliated single-layer MoS<sub>2</sub> are indicated with dashed lines for comparison. In (b) and (c), the error bar represents one standard deviation.

MoS<sub>2</sub> [47]. Additionally, the measured values of  $\Delta k$  for the CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C were 21.2 cm<sup>-1</sup>, 21.0 cm<sup>-1</sup>, and 20.5 cm<sup>-1</sup>, respectively,

which agrees with those of CVD-grown single-layer MoS<sub>2</sub> [32, 49]. These Raman measurement results, along with the AFM data shown in Fig. 1(b), show that single-layer MoS<sub>2</sub> formed on the substrate for both the mechanical exfoliation and CVD processes. In addition, the larger values of  $\Delta k$  for the CVD-grown MoS<sub>2</sub> suggest that the CVD-grown MoS<sub>2</sub> may have inferior crystalline qualities compared to the mechanically exfoliated MoS<sub>2</sub> [32, 49].

To further evaluate the crystalline qualities of mechanically exfoliated and CVD-grown MoS<sub>2</sub>, the full width at half maximum (FWHM) of the  $E_{2g}^1$  peak was also obtained from the Raman data [49], as shown in Fig. 2(c). The FWHM of the  $E_{2g}^1$  peaks for the mechanically exfoliated MoS<sub>2</sub> was 3.4 cm<sup>-1</sup>. Further, as shown, the FWHM of the  $E_{2g}^1$  peaks for the CVD grown MoS<sub>2</sub> at 800 °C and 850 °C agreed with that of the mechanically exfoliated MoS<sub>2</sub>. However, the FWHM of the  $E_{2g}^1$  peak for the CVD-grown MoS<sub>2</sub> at 900 °C was significantly larger than that of the mechanically exfoliated MoS<sub>2</sub>. This result further indicates that CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C have better crystalline qualities than the CVD-grown MoS<sub>2</sub> at 900 °C [36, 49].

After characterization of the MoS<sub>2</sub> specimens using an optical microscope, AFM, and Raman spectroscopy, the adhesion and friction characteristics were determined from the force-distance curves and friction loops, respectively. Figure 3 presents examples of the force-distance curves and average adhesion forces between the Si tip used in this work and the MoS<sub>2</sub> specimens, obtained before and after friction loop measurements. As shown in Fig. 3(a), the adhesion forces between the Si tip and CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C were about 16 nN, which agreed with values found between the Si tip and mechanically exfoliated MoS<sub>2</sub>. However, the adhesion force between the Si tip and CVD-grown MoS<sub>2</sub> at 900 °C was slightly larger than in the other cases. As shown in Fig. 3(b), the average adhesion forces of mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C were 17 ± 2 nN, 16.0 ± 0.3 nN, 16.1 ± 0.7 nN, and 19.8 ± 0.4 nN, respectively. It was hypothesized that interactions with the Si tip might have been enhanced due to imperfections in the crystalline structure, which resulted in a relatively large adhesion force of the CVD-grown single-layer



**Fig. 3** (a) Examples of force-distance curves and (b) average adhesion forces of the mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C. In (b), the adhesion forces obtained before and after friction loop measurements are compared. In (b), the error bar represents one standard deviation.

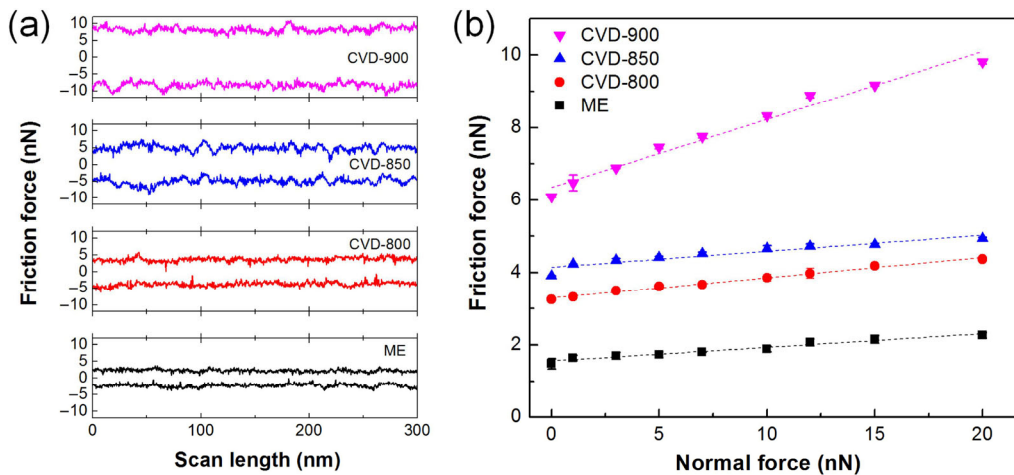
MoS<sub>2</sub> at 900 °C. In addition, the adhesion force for the mechanically exfoliated MoS<sub>2</sub> slightly decreased after the friction loop measurements. A ripple structure may have been formed due to inhomogeneous interaction between MoS<sub>2</sub> and substrate during mechanical exfoliation, which in turn caused the local differences in frictional and material properties [18, 50]. This local difference in materials properties of the mechanically exfoliated MoS<sub>2</sub> might have caused the difference in adhesion force before and after friction loop measurements, as shown in Fig. 3(b). However, because the CVD-grown MoS<sub>2</sub> may exhibit larger adhesion to the substrate compared to the mechanically exfoliated MoS<sub>2</sub> [51], the effect of ripple structures on the adhesion of CVD-grown MoS<sub>2</sub> was expected to be relatively small. This small effect may be responsible

for the good agreement between the adhesion forces of CVD-grown MoS<sub>2</sub> before and after friction loop measurements. In addition, the data shown in Fig. 3 suggests that the tip wear that occurred during friction loop measurements was negligible.

Figure 4(a) shows examples of friction loops for the mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C obtained under 10 nN normal force. The friction force of the mechanically exfoliated MoS<sub>2</sub> was about 2 nN under this normal force of 10 nN. However, the friction forces of the CVD-grown MoS<sub>2</sub> were significantly larger than that of the mechanically exfoliated MoS<sub>2</sub>, increasing from 3.7 nN to 8.3 nN as temperature increased from 800 °C to 900 °C with 10 nN of applied normal force.

Variations in the friction force of mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C with respect to the normal force are shown in Fig. 4(b). In general, the friction force increased as the normal force increased, as expected. However, a nonlinear relationship between the friction force and normal force [52] was not clearly observed in this work. Figure 4(b) shows that force of the single-layer MoS<sub>2</sub> prepared using mechanical exfoliation increased from 1.5 nN to 2.3 nN when the normal force increased from 0 to 20 nN. As shown, the friction forces of the CVD-grown MoS<sub>2</sub> were significantly larger than those of the mechanically exfoliated MoS<sub>2</sub>, which increased with increasing CVD temperature. The friction forces of the CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C were, respectively, 2.0, 2.4, and 4.2 times larger than that of the mechanically exfoliated MoS<sub>2</sub>. The friction forces under a zero normal force were also the greatest for the CVD-grown MoS<sub>2</sub> at 900 °C. Furthermore, the increases in friction force with increasing normal force were relatively gradual for the mechanically exfoliated MoS<sub>2</sub> and the CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C, whereas the increase was much more significant for the CVD-grown MoS<sub>2</sub> at 900 °C.

Many factors, such as the number of layers [14, 17], surface topography [20, 23], interactions with the substrate [21], and crystalline orientation [18] can affect the friction characteristics of two-dimensional materials, in addition to the experimental and environmental conditions [24]. The dependence of

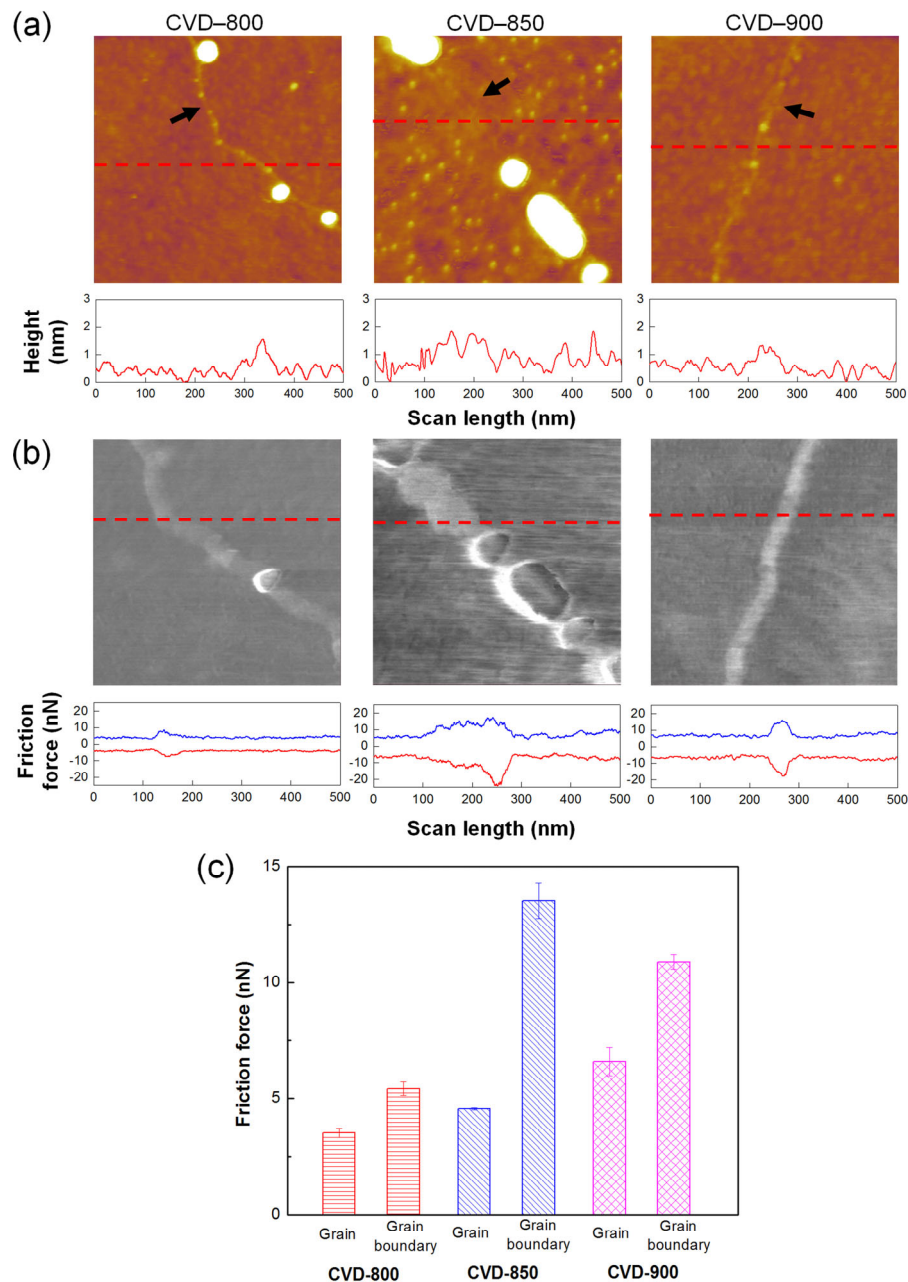


**Fig. 4** (a) Examples of friction loops and (b) variations in friction force of the mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C with respect to the normal force. In (a), the friction loops were obtained under 10 nN normal force. In (b), the dashed lines indicate the linear curve fits and the error bar represents one standard deviation.

MoS<sub>2</sub> friction characteristics on the surface topography is not clearly indicated in Figs. 1 and 4. This lack of correlation might have been due to the surface topographies of the MoS<sub>2</sub> specimens being too homogeneous to affect the friction characteristics. In addition, the effect of crystalline orientation on the friction characteristics was unlikely to appear in the data presented in Fig. 4, considering that the frictional anisotropy due to crystalline orientation is greater at a lower normal force [18]. However, the Raman data in Fig. 2 suggest that the larger friction characteristics of CVD-grown MoS<sub>2</sub> compared to mechanically exfoliated MoS<sub>2</sub> was likely associated with the crystalline imperfections of the CVD-grown MoS<sub>2</sub>. The crystalline qualities of CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C were relatively close to those of the mechanically exfoliated MoS<sub>2</sub>. This can explain why the friction characteristics of CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C were relatively close to those of the mechanically exfoliated MoS<sub>2</sub>. However, the crystalline qualities of MoS<sub>2</sub> grown at 900 °C were significantly lower than those of the mechanically exfoliated MoS<sub>2</sub>, which may be responsible for the relatively high friction characteristics. It should also be noted that the large friction characteristics of the CVD-grown MoS<sub>2</sub> at 900 °C may be related to the large adhesion shown in Fig. 3.

The CVD-grown MoS<sub>2</sub> may readily contain grain boundaries because isolated flakes of MoS<sub>2</sub> were

merged into a continuous film during the CVD process. Therefore, understanding the friction characteristics at grain boundaries of CVD-grown MoS<sub>2</sub> is important. Figure 5 shows the AFM topographic and friction force images obtained under the intermittent contact and contact modes, respectively, over the grain boundaries of the CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C. The cross-sectional height profiles and friction loops are also compared in Fig. 5. The friction force images were obtained under 3 nN normal force concurrently with the friction loop. The grain boundaries can be clearly observed in the topographic images. In addition, the cross-sectional height profiles show that the heights at the grain boundaries were slightly larger than those at the grains. The friction force images and friction loops clearly show that the friction forces at the grain boundaries were significantly larger than those at the grains. However, the friction forces on the left and right sides of the grain boundaries were quite similar to each other, indicating that there was an insignificant effect of crystalline orientation on friction of the CVD-grown MoS<sub>2</sub> specimens. The friction forces under 3 nN normal force at the grains and grain boundaries of the CVD-grown MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C are shown in Fig. 5(c). Compared to the grains, the friction at the grain boundaries increased by factors ranging from 1.5 to 3.0. Defects at the grain boundary may lead to changes in the frictional properties, similar to the



**Fig. 5** (a) AFM topographic images and (b) friction force images of the CVD-grown single-layer MoS<sub>2</sub> at 800 °C, 850 °C, and 900 °C, and (c) comparison of the friction forces at the grains and grain boundaries of the CVD-grown MoS<sub>2</sub>. The topographic images were obtained with the intermittent contact mode, and the friction force images were obtained using the AFM contact mode under 3 nN normal force. The red dashed lines in (a) and (b) indicate the locations of the cross-sectional height profiles and friction loops, respectively. In (a), the grain boundaries are indicated with black arrows. In (c), the error bar represents one standard deviation.

optical and electrical properties [34, 35]. This outcome suggests that the CVD growth temperature for MoS<sub>2</sub> should also be optimized for grain boundary density to achieve low friction characteristics, considering that CVD growth temperature often affects grain size, as shown in Fig. 1.

According to the experimental results, the CVD-grown single-layer MoS<sub>2</sub> showed greater friction than the mechanically exfoliated single-layer MoS<sub>2</sub>, which may be associated with crystalline imperfections in the CVD-grown MoS<sub>2</sub>. This outcome indicates that the growth of the single-layer MoS<sub>2</sub>, with high



crystalline quality, is preferred when trying to achieve low friction characteristics. The friction force measurement results also suggest that the optimal CVD growth temperature is 800 °C based on tribological concerns. However, it should be noted that the frictional properties strongly depend on the test parameters, such as contact pressure, sliding speed, and environmental conditions. In addition, the MoS<sub>2</sub> crystalline structure and quality can be significantly affected by the deposition method and conditions. From this perspective, the results of this work cannot be generalized to all types of single-layer MoS<sub>2</sub>. Nevertheless, the outcomes of this work are valid for similar types of MoS<sub>2</sub> grown using the CVD process. Furthermore, the results presented in this work are expected to aid in a more comprehensive and fundamental understanding of the frictional properties of single-layer MoS<sub>2</sub>.

## 4 Conclusions

In this work, the friction characteristics of mechanically exfoliated single-layer MoS<sub>2</sub> and CVD-grown MoS<sub>2</sub> at three different temperatures were systematically investigated using AFM. The mechanically exfoliated and CVD-grown MoS<sub>2</sub> specimens were also examined using an optical microscope, AFM, and Raman spectroscopy to understand the surface characteristics and crystalline qualities of the specimens. The surfaces of the mechanically exfoliated and CVD-grown MoS<sub>2</sub> were generally flat and smooth. The Raman data showed that the crystalline qualities of CVD-grown MoS<sub>2</sub> at 800 °C and 850 °C were relatively close to those of mechanically exfoliated MoS<sub>2</sub>. However, the crystalline qualities of the CVD-grown MoS<sub>2</sub> at 900 °C were lower compared to those of the mechanically exfoliated MoS<sub>2</sub>. The CVD-grown MoS<sub>2</sub> exhibited greater friction than the mechanically exfoliated single-layer MoS<sub>2</sub>, which might have been associated with the crystalline imperfections in CVD-grown MoS<sub>2</sub>. In addition, the friction of the CVD-grown MoS<sub>2</sub> increased as the CVD growth temperature increased. In terms of the frictional properties, the optimal growth temperature was 800 °C for the CVD process used in this work. Furthermore, the friction at the grain boundaries was larger than at the grain interior by factors of 1.5 to 3.0, which might have been due

to defects at the grain boundary. The outcomes of this work are expected to advance the fundamental understanding of intrinsic friction characteristics of single-layer MoS<sub>2</sub>, and elucidate the feasibility of single-layer MoS<sub>2</sub> as protective or lubricant layers for micro- and nano-devices.

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