ABSTRACT: Encouragingly, a lot of research studies have demonstrated that two-dimensional (2D) nanosheets applied as an additive in oils show preferable friction-reducing and wear resistance performance. However, the current issue was that an elusive way could be adopted to probe the structure–activity relationship between the structure and tribological properties of bulk layered materials due to the structural evolution during friction testing. In this study, we studied the structure–activity relationship between the structure and tribological properties of bulk layered materials (graphite, h-BN, WS2, and MoS2) by an in situ four-ball friction tester. The morphological and structural changes of the layered materials after in situ four-ball-milling were detected by a series of characterizations. This study revealed the friction-induced nanostructural evolution behaviors of bulk layered materials by a four-ball mode.

INTRODUCTION

Most of the energy consumption and material loss along with growing transportation and other industries were ascribed to friction and wear, as a result accelerating the emission of CO2 and other harmful gases. Currently, the most effective way to control or reduce friction and wear is to use a lubricant with good friction properties.1 The two-dimensional (2D) layered materials as lubricant additives were capable of forming protective films or sliding layers on the contact surface, which were very effective in reducing friction and wear.2 Lately, the bulk layered materials (e.g., graphite, MoS2, WS2, and h-BN) have extensively been used as lubrication additives to improve the friction and wear properties of lubricants.3,4 Due to the intrinsically low friction properties of the 2D layered nanosheets structures, they were desirable in many engineering applications, where friction coefficients were reduced to 0.07–0.27.5–8 The proposed mechanism of the friction-reducing performance, which has been revealed to be a result of a durable boundary film on the rubbing surfaces to prevent metal-to-metal contact, relied on sliding between the layers because of weak van der Waals forces.9–12 Consequently, the structure of 2D layered nanosheets played an intriguing role in the lubrication properties,10,13 thus accounting for an unknown relationship between the structure and friction properties.14,15 The reports showed that graphene with a certain amount of native surface dislocation, vacancies, or interlayer corrugation presented a higher friction coefficient than the defect-free graphene.16,17 Fundamentally, the Raman intensity of the D peak and the \( I_D/I_G \) ratio of the defect-free graphene debris after use increased, suggesting that the defects and disorders emerged in some local regions of graphene.18,19 However, after being tested in the duration of the friction process, the \( I_{2D}/I_G \) ratio of graphene decreased, indicating that graphene was stacked to form thick sheets. This gave rise to the formation of a friction film that separated the two contact surfaces.20,21 Even at the nanoscale, the direct observation of the tribolayers formed on the friction interfaces by high-resolution transmission electron microscopy (HRTEM) also suggested the ordering evolution of graphene.22–24 Hence, the friction-induced structural change for 2D layered nanosheets was generally identified. In other cases, many graphite-like layered materials with an ordered structure also achieved lower friction.25 For producing high-quality 2D layered nanosheets, many exfoliation methods have sprung up in the past decade.26–28 Among these methods, the exfoliation by ball-milling has successfully produced a catalog of 2D layered nanosheets (graphene, MoS2, boron nitride nanosheets
In a liquid phase, we envisioned a study method to reveal the relationship between the structural change and the friction coefficient of 2D layered bulk materials on the basis of a four-ball friction tester.

Through in situ testing of the friction performance in a four-ball tester, we characterized the structure and morphology of the bulk layered materials at different friction times and discussed the relationship between the structural change and friction performance.

**RESULTS AND DISCUSSION**

Figure 1 provides the particle size distributions of four two-dimensional materials dispersed in glycerol. As a result of three-time measurements of each two-dimensional material, it is found that the bulk particles of the four two-dimensional materials are uniformly distributed in glycerol, and their average particle sizes are approximately 788, 244, 507, and 587 nm, respectively.

Figure 2 shows the friction coefficient curves and scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images of four two-dimensional materials before and after ball-milling. It can be seen from Figure 1A that the coefficient of friction (COF) of graphite gradually increases to a maximum of 0.1 within 0−2600 s due to the aggregation and shearing of graphite at the friction interface. Subsequently, the COF gradually decreases to 0.042 in the range of 2600−21 600 s, showing a decrease by 58% compared to the maximum. This phenomenon is attributed to the antifriction effect of graphite. In general, a horizontal slip of low shear force occurs between the layers of graphite with a high-speed sliding of the friction interface. Correspondingly, the SEM and TEM images show that the pristine graphite particles exhibit a typical bulklike structure with a lateral size of 2−4 μm (Figure 2A1,A2). After undergoing ball-milling exfoliation in the four-ball mode, the bulk graphite is significantly converted into a sheetlike structure, accompanied by a 58% reduction in the friction coefficient and a decline to 0.5−1.2 μm in the average size (Figure 2A3,A4). Interestingly, the same results are found in the other three layered materials (Figure 2B−D). In contrast to the rigid and thick raw materials (Figure 2B1,B2,D1,D2), the edges and surfaces of ball-milled materials seem to be curly and coarse (Figure 2B3,B4,D3,D4), suggesting a rather low thickness. It is possible that, as the aid of continuous shearing, the van der Waals forces in the direction perpendicular to the surfaces were overcome, leading to the warping and exfoliation of bulk sheets into nanosheets.

It is noteworthy that except for WS2, the other three materials exhibit a significant reduction in both size and thickness. The WS2 bulk material has not only larger size and thickness but also stronger van der Waals forces in the interlayer, thereby accounting for the difficulty in exfoliation in the four-ball mode (Figure 2C1−C4).

To further understand the structural evolution of the bulk layered materials under friction testing, we conducted HRTEM analysis (Figure 3) for the bulk layered materials before and after ball-milling. Through the in situ ball-milling, we can obtain several to dozen layers of nanosheets from raw materials. The reason is that, with the aid of continuous shearing, the van der Waals forces in the direction perpendicular to the surfaces of 2D materials are overcome, thus leading to the peeling of raw materials into nanosheets. This result further confirms the structural transformation from the bulk layered materials to few layers during sliding. However, the ball-milled h-BN and WS2 show obvious disorders and defects in their structures, which means that highly ordered or less exfoliated bulk layered materials are prone to generating uncertain defects by friction. In all, the results evidently reveal that friction-induced nanostructural evolution occurs.

Figure 4 shows the X-ray diffraction (XRD) and Raman spectra of the bulk layered materials and ball-milled layered samples. For the four ball-milled materials, the XRD
characteristic peaks show weaker intensity and a slight blue shift, suggesting the enlargement of the interlayer spacing and the indication of a weakened $\pi-\pi$ stacking interaction.\textsuperscript{38,39}

The Raman spectra before and after ball-milling are shown in Figure 3B. Compared to the Raman spectra of graphite, the D band of ball-milled graphite at $\sim1350$ cm$^{-1}$ is attributed to the

Figure 2. Friction coefficient curve, corresponding SEM and TEM images of ball-milled graphite (A1–A4), ball-milled h-BN (B1–B4), ball-milled WS$_2$ (C1–C4), and ball-milled MoS$_2$ (D1–D4) at different friction stages.
The defect-induced breathing mode of sp³ rings and the G band at \( \sim 1582 \text{ cm}^{-1} \) is attributed to the E\(_{2g}\) mode of sp²-hybridized carbon bonds.\(^{40}\) Meanwhile, the enhanced D peak indicates an increase in the order degree of the exfoliated graphite.\(^{41}\) For h-BN, the strong peak at 1365 cm\(^{-1}\) is attributed to the interlayer E\(_{2g}\) mode of h-BN. After ball-milling, the E\(_{2g}\) peak of the as-

Figure 3. HRTEM images of ball-milled graphite (A1, A2), ball-milled h-BN (B1, B2), ball-milled WS\(_2\) (C1, C2), and ball-milled MoS\(_2\) (D1, D2) at the lowest friction coefficient.
exfoliated h-BN shows a slight blue shift and a decrease in strength, indicating an increase in disorder and a decrease in the number of layers.42,43 For WS2, the intensity ratios of the two peaks decrease, indicating an increase in structural disorder. On the contrary, the intensity ratio of the two peaks of MoS2 increases, indicating that the structure is more ordered. This is consistent with the results of HRTEM.

Figure 5 shows the Fourier transform infrared (FT-IR) and UV−vis spectra of the bulk layered materials and ball-milled layered samples. Apparently, the UV−vis characteristic peak of the ball-milled layered materials shifts to a higher peak position, indicating a reduced layer number (Figure 5A).44−46 As shown in the FT-IR spectra of Figure 4B, for ball-milled graphite, the obvious augmentation of the band at 3125 cm−1 is associated with the stretching vibrations of OH47 and that of the band at 1748 cm−1 arises from the asymmetrical stretching of C=O groups, and the peaks in the region of 1500 cm−1 correspond to OH vibrations and single bond energy between C and O stretching of C−O−C and C−OH, corroborating the increase of oxygen content after ball-milling.48 For h-BN, there is no significant change in the infrared spectrum before and after ball-milling. In the infrared spectra of WS2 and MoS2, there appears a decrease in the intensities of the W−S bond and Mo−S bond but an increase in the intensity of the S−O bond. In general, the oxygen contents of several materials after ball-milling significantly increases due to oxidation of the sheet material during the rubbing process.

The wear behavior of different bulk layered materials can be illustrated clearly by the morphology of wear scars at the lowest friction coefficient as shown in Figure 6. Among these wear scars, those of h-BN and WS2 exhibit a deeper and wider wear region (Figure 6B,C) than those of graphite and MoS2 (Figure 6A,D). For MoS2, the abundant active elements such as Mo and S play an important role in reducing the wear and increasing the load capacity.9,50 However, the worn surface of graphite shows smaller wear, which is ascribed to the flexible layered graphite filling the worn grooves by exfoliating off few graphene nanosheets and forming stable transfer films on the worn surface.51−53 Therefore, it can be inferred that the relationship between the structural evolution and friction performance can be revealed by studying the materials before and after ball-milling.

The Raman spectra of wear scar areas are shown in Figure 6. It can be seen that a more noticeable and stronger D peak appears on the friction interface lubricated by the ball-milled graphite (Figure 7A).54 Similarly, the Raman signals of the exfoliated h-BN and WS2 are found on the worn surfaces. These results demonstrate that the exfoliated nanosheets can be filled into the groove of the worn surface in ball-milling.55−57 Therefore, it can be concluded that ball-milling plays a key role in the structural evolution of bulk layered materials, and the coefficient could reflect the structural change in bulk layered materials at the friction interface to some extent. That is to say, the originally integrated structure makes a great contribution to the better lubrication properties.

**CONCLUSIONS**

In this study, we designed a method to probe the in situ structure−activity relationship between structural evolution during ball-milling and the friction coefficient in the four-ball mode. The results showed that the change of friction coefficient in testing was connected to the structural evolution of the bulk layered materials.

**EXPERIMENTAL SECTION**

**Ball-Milling Procedure.** In a typical experiment, 0.1 g of raw material (graphite, h-BN, WS2, MoS2) Sinopharm...
Chemical Reagent Co., Ltd., 99%) and 15 mL of glycerin (Sinopharm Chemical Reagent Co., Ltd., 98.5%) were poured into a container and sonicated for 2 h to obtain a uniform dispersion. Five milliliters of the mixture was then transferred into a stainless steel oil canister in a four-ball friction machine. The ball-milling test of the samples was carried out by a four-ball machine (MMW-1, Jinan Chenda Co., Ltd.). The testing was conducted according to the ASTM D4172 standard method (1200 rpm, 100 N load, and testing duration of 6 h). After the completion, the collected product was washed with absolute ethanol and water and then centrifuged for 15 min at 500 rpm. After centrifugation, the top 80% of the supernatant was pipetted off and the ball-milled layered material was characterized. The illustration of the relationship studied between the coefficient of friction and structure of the bulk layered materials is described in Scheme 1.

Figure 6. Three-dimensional (3D) topography images and 2D profiles across the wear tracks for flat specimens after wear tests with graphite (A), h-BN (B), WS₂ (C), and MoS₂ (D) (100 N, 1200 rpm, 6 h).

Characterizations. The morphologies and sizes of the as-prepared samples were observed by a field scanning electron microscope (S-4800II, HITACHI, Japan) equipped with an energy-dispersive spectrum (EDS) and a transmission electron microscope (Tecnai 12, Philips, Netherlands). High-resolution transmission electron microscopy (HR-TEM) was conducted on a Tecnai G2F30S-TWIN field emission transmission electron microscope. The phase composition of the as-prepared samples was investigated using a D8 advance X-ray diffraction instrument (XRD, Bruker AXS, Germany). Raman spectra were investigated (using an In Via Raman spectrometer, Renishaw, Britain). Fourier transform infrared spectroscopy (FT-IR) signals were recorded on a Cary 610/670 micro infrared spectrometer (Varian). Further, the ultraviolet and visible (UV−vis) spectra were investigated by a Cary 5000 spectrophotometer (Varian).
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Notes

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