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Research Article

Study of Tribological Properties of Nanolamellar WS_2 and MoS_2 as Additives to Lubricants

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This work was aimed at studying the tribological properties of nanolamellar tungsten and molybdenum disulfides produced from nanosized W and Mo nanopowders by self-propagating high-temperature synthesis. The prepared WS_2 and MoS_2 powders were examined by scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential thermal analysis (DTA). For tribological tests, oil-based lubricants added with nanolamellar tungsten and molybdenum disulfides were prepared. The tribological tests show that the friction coefficient of the nanolamellar powders is lower than that of commercial powder ($\mu_{\min} = 0.024$ and 0.064, resp.). It is also found that the oil-based lubricants with nanolamellar disulfide additives display higher antifriction and antiwear properties compared to commercial powder.

1. Introduction

Nowadays, there is a trend among tribologists to investigate peculiar lubricious effects of nanostructured materials. Particular attention is given to nanocomposite lubricants. One of the most interesting challenges is to drastically decrease the friction coefficient between surfaces and to improve the antiwear action of nanosized particles of sandwich-like structures, for example, transition metal chalcogenides.

Composite lubricants based on nanostructured metal dichalcogenides are promising materials due to their excellent tribological properties (low coefficient of friction, antiwear action). Previously, Jamison and Cosgrove [1] correlated the lowest coefficient of friction to the axial ratio c_0/na_0 which might not exceed 1.87. For 2H-MoS_2 and 2H-WS_2, the axial ratio is equal to 1.95 and 1.96, respectively. Under standard conditions, tungsten and molybdenum disulfides have almost the same coefficient of friction (up to 0.02), but WS_2 reveals better thermal and oxidation stability than MoS_2 [1]. Recent interest of scientists has been focused on studying the superlubricity of nanomaterials. So, Miura and Kamiya [2] supposed that Amontons-Coulomb law should hold for molybdenum disulfide nanoflakes. They found that the coefficient of friction decreased down to 0.003 between two MoS_2 surfaces along the [1010] direction of the MoS_2 (0001) plane. Lamellar metal chalcogenides (Me_xS_y, Me_xSe_y, and Me_xTe_y) can be used as solid lubricants and additives in oil and greases [3–6] and also as various coatings (on ceramics, glass, polymers, and metals) [7–9]. It has been also reported that MoS_2 thin films were successfully prepared by ionic layer adsorption and reaction method [10]. The WS_2 nanoparticles can be incorporated into a coating matrix to form nanocomposite coatings in order to affect its tribological properties [11].

Nanostructured tungsten and molybdenum disulfide powders and thin films are conventionally produced via direct reaction between nanosized metal and elementary sulfur [12], sulfurization of nanostructured molybdenum oxide [13], sonochemical treatment of metal salt and sulfur mixture [14], dispersion of coarse-grained MoS_2 [15], and self-propagating high-temperature synthesis (SHS) with tetrathiotungstate precursor formation [16]. The main disadvantages of the above methods are low productivity, impurities contained in resulting substances, and simultaneous presence of different sulfide phases (2H– and 3R–). One of the most interesting techniques to produce WS_2 fullerene-like nanoparticles has been reported in [17]. The nanoparticles were synthesized by a solid-gas reaction between WO_3 and H_2S in a reducing atmosphere. According to the authors, the tribological tests demonstrated that the coefficient of friction for the prepared particles having a mean size of 120 nm was 0.03 at a load of 150 N. The authors of [18] have tried to compare tribological behavior of MoS_2 and WS_2 fullerene-like particles, nanotubes, and platelets. It has been reported that their morphology did not significantly affect the coefficient of friction. The question on the most efficient tribological material among different types of nanostructured molybdenum and tungsten disulfides is still under discussion.

In this study, we examined the antiwear properties of nanolamellar WS_2 and MoS_2 powders produced by SHS from tungsten or molybdenum nanopowders and elementary sulphur in an inert atmosphere. The use of tungsten and molybdenum nanopowders in self-propagating high-temperature synthesis ensures the nanolamellar structure of the obtained disulfides.

2. Experimental

2.1. Synthesis of Nanolamellar MoS_2 and WS_2 Powders. Nanolamellar tungsten and molybdenum disulfides were synthesized by self-propagating high-temperature synthesis using nanosized tungsten and molybdenum powders and pure elementary sulphur. The nanosized tungsten and molybdenum powders were prepared by electrical explosion of wires (EEW) in argon. The EEW method was used due to its high efficiency in producing a large variety of metals and compounds [19, 20]. The use of tungsten and molybdenum nanopowders was dictated by their high specific surface area and reactivity. This ensured high interaction rates and stabilization of nanolamellar WS_2 and MoS_2 particles in the final product.

The mixture obtained from nanodispersed metal powder and pure elementary sulfur was pressed into cylindrical pellets of diameter 32 mm. Self-propagating high-temperature synthesis of nanolamellar tungsten and molybdenum disulfides was realized on the experimental setup described elsewhere [21]. The article describes physicochemical properties and structure of the obtained nanolamellar particles. The setup comprised a hermetic chamber rated at a pressure limit of 50 atm. The chamber was equipped with units for working gas intake and offtake and with a pellet holder. Combustion of the sample was initiated with a nichrome filament. The combustion temperature was measured with a tungsten-rhenium thermocouple, the hot junction of which was placed into the pellet bore. The thermocouple cold ends were connected to an oscilloscope. The combustion process was controlled through an observation port.

The synthesized products were easily disaggregated silvery-black sinters. They were milled and washed out of sulfur traces in hexane and simultaneously treated in an ultrasonic bath. After drying, the synthesized powders were analyzed by XRD (Shimadzu XRD-7000 diffractometer, CuK_α radiation), TEM (JSM-7500FA, JEOL), and DTA (Q600 SDT Simultaneous DSC-TGA).

2.2. Sample Preparation for Tribological Tests. For tribological tests, lubricants based on nanolamellar tungsten and molybdenum disulfides were prepared. The solid lubricants were WS_2 and MoS_2, washed out of sulphur traces and treated in an ultrasonic bath. Before measurements of the coefficient of friction, oil with nanolamellar powders was prepared as follows. For studying the effect of nanolamellar and commercial disulfides, base oil M8V (Russian oil designation standard) was used. According to the supplier, the kinematic viscosity of the M8V oil was 7.5–8.5 mm^2/s at 100°C. Its density was 905 kg/m^3 at 20°C. The base oil was mixed with 5 wt.% of nanolamellar tungsten and molybdenum disulfide additives and 10 wt.% of dispersing agent. Before tribological study, the mixture of base oil and disulfide was treated in an ultrasonic bath for 1 hour.

The coefficient of friction of the lubricants based on nanolamellar tungsten and molybdenum disulfides was measured with a "ball-on-disk" PC-Operated High-Temperature Tribometer (THT-S-AX0000, CSEM). The wear was studied using a noncontact profilometer (Micro Measure 3D Station, STIL, France). Medium-carbon steel disks of diameter 30 mm, height 4 mm, and surface roughness $R_a = 60$ nm were used as the body, and a hard alloy ball of diameter 3 mm was used as the counterbody. The normal load was 5 N, the temperature was 25°C, the linear speed was 5 cm/s, and the wear scar radius was 3 mm.

After tribological tests, the disk surface was cleaned with acetone and the wear scars were measured with an optical microscope. The wear scar area equal to 2 mm × 1 mm was scanned and the volume wear was calculated by six points using profilometer software. The scar roughness was also measured.

3. Results and Discussion

3.1. Physicochemical Properties of Nanolamellar MoS_2 and WS_2 Powders. According to the XRD analysis, the main phases are 2H-WS_2 and 2H-MoS_2. The obtained powders, as evidenced by SEM images (Figures 1(a) and 1(b)), are agglomerates of lamellar particles of thickness 50–150 nm. Obviously, most of the particles have a crystallized hexagonal shape and their width ranges from 100 nm to several microns. The lamellae are rather extended and are 20–40 nm wide. Some lamellar particles possess a multilayer structure, being indicative of their layer-by-layer condensation from liquid on cooling the reacted mixture. XRD analysis showed that the main phases are MoS_2 and WS_2, respectively (Figures 1(c) and 1(d)).

Thermal and oxidation stability at high temperature is a significant property for lubricants. It is well known that low thermal and oxidation stability of lubricating materials can limit their application at high temperatures [22]. The thermal behavior of the obtained nanolamellar tungsten and molybdenum disulfides was studied in air using a thermoanalyser at

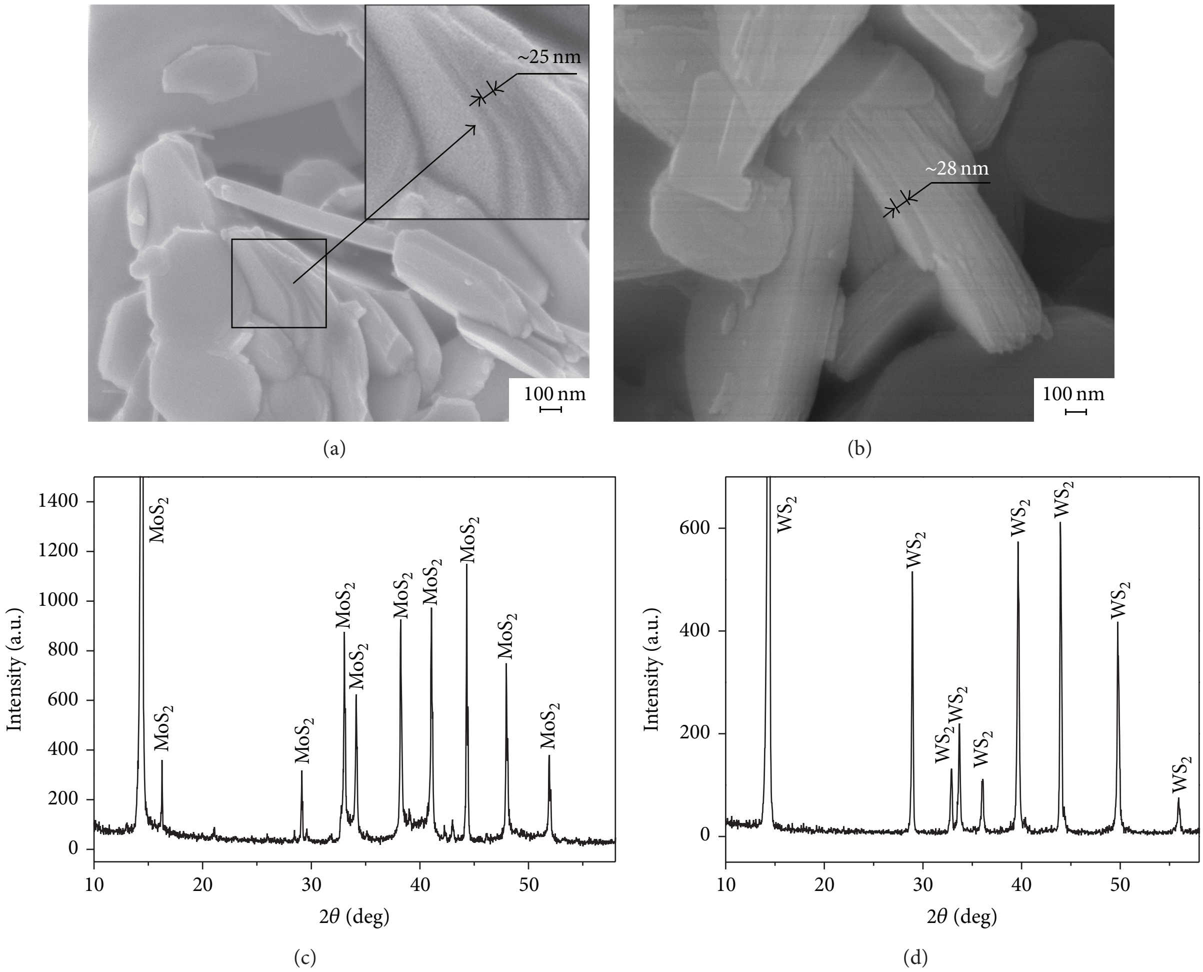

Figure 1: SEM images and XRD pattern for tungsten disulfide (a, c) and molybdenum disulfide (b, d).

a heating rate of 10°C/min. Heat flow and gravimetric curves for WS_2 and MoS_2 are shown in Figure 2.

The heating of tungsten disulfide (Figure 2(a)) is characterized by a negligible leeway of the thermogravimetric curve (curve 1) and by a small endothermic effect (curve 2) of sulfur trace melting. Nanolamellar tungsten disulfide is stable up to 450°C. Its further heating up to 700°C causes a small weight loss (4%) due to a negligible exothermic effect. This effect can be due to prime disulfide decomposition into tungsten and sulfur with subsequent oxidation of the latter and sulphur oxides loss. The same is observed for nanolamellar molybdenum disulfide, though the sample weight loss is higher (7%). At the same thermal stability temperatures above 450°C, nanolamellar MoS_2 is thus decomposed more intensively than nanolamellar WS_2. Similar thermochemical behavior and stability are also characteristic of regular molybdenum and tungsten disulfides. This fact allows us to expect thermally stable nanolamellar disulfides with novel tribological performances.

3.2. Measurement of the Coefficient of Friction.

Hexagonal molybdenum disulfide MoS_2 is a much used solid lubricant, whereas tungsten disulfide WS_2 is frequently used as an antifriction additive stable at higher temperatures (up to 500°C). Some recent publications report that nanocrystalline tungsten and molybdenum disulfides with spherical particles have better antifriction properties compared to those with ordinary microparticles. However, we consider that nanolamellar tungsten and molybdenum disulfides can greatly improve the antifriction properties of materials.

Tribological tests of the as-prepared tungsten and molybdenum disulfides were performed at room temperature as described above. In order to compare the tribological behavior of nanolamellar disulfides with that of regular powders, commercial molybdenum disulfide powder was also tested as a reference. The coefficient of friction for nanolamellar tungsten disulfide at room temperature is unstable ($\mu_{\text{min.}}$ = 0.035, $\mu_{\text{aver.}}$ = 0.051), whereas that of molybdenum disulfide (Figure 4(a)) is lower and more stable with time ($\mu_{\text{min.}}$ = 0.024, $\mu_{\text{aver.}}$ = 0.028). The significant instability of the coefficient of friction for nanolamellar tungsten disulfide in tribological testing is probably due to stronger van der Waals interaction between sulfur layers, compared to nanolamellar molybdenum disulfide. The friction coefficient of commercial

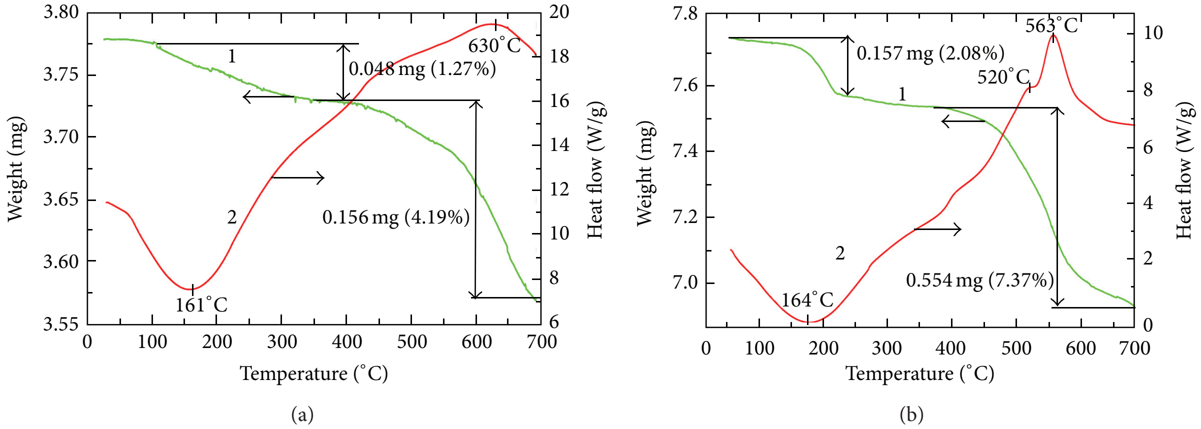

FIGURE 2: DSC-TGA results for nanolamellar WS_2 (a) and MoS_2 (b): 1—mass loss; 2—heat flow.

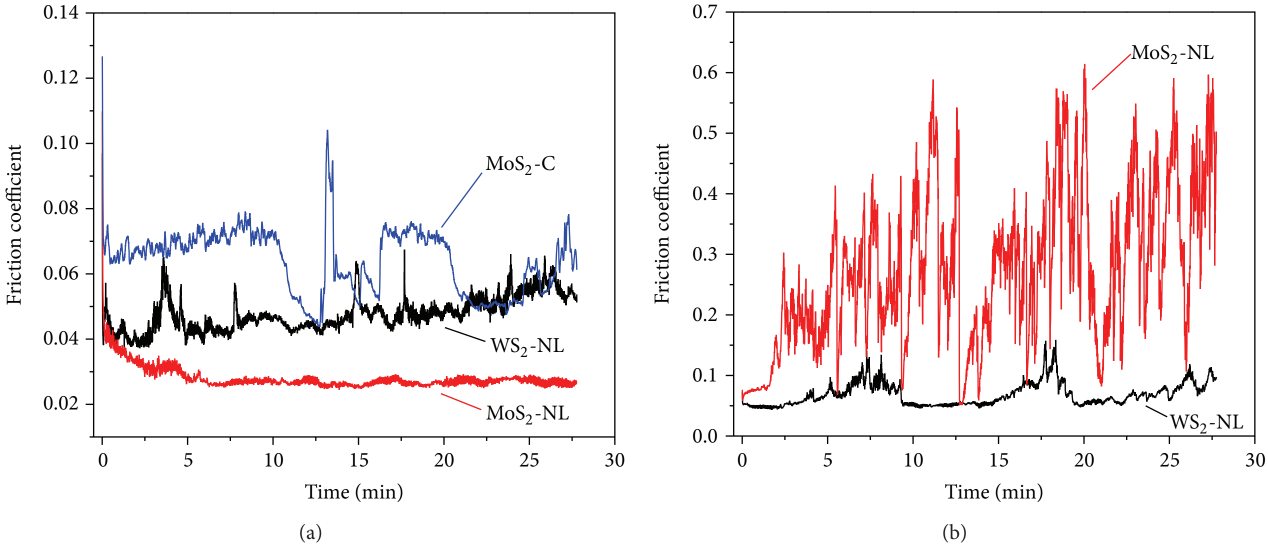

FIGURE 3: Friction coefficient of nanolamellar (NL) WS_2 and MoS_2 and commercial (C) micron-sized MoS_2 powder at 25°C (a) and 400°C (b).

MoS_2 displays more intense fluctuation, and its average value is equal to 0.064 (Figure 3(a)).

Supposedly, the higher stability of the coefficient of friction for the MoS_2-NL sample can be associated with the formation of a durable tribofilm due to efficient rubbing of nanolamellar MoS_2 particles.

At higher temperatures (400°C), significant instability and the high coefficient of friction ($\mu_{\text{min.}} = 0.051$, $\mu_{\text{aver.}} = 0.274$) are characteristics of molybdenum disulfide (Figure 3(b)), whereas the coefficient of friction for tungsten disulfide is lower and more stable with time ($\mu_{\text{min.}} = 0.043$, $\mu_{\text{aver.}} = 0.068$). These data agree with DTA data on the synthesized powders. At temperatures above 400°C, nanolamellar molybdenum disulfide exhibits lower stability and splits into metal and sulfur more intensively than tungsten disulfide. The decomposition of molybdenum disulfide greatly increases the coefficient of friction due to metal formation on disulfide decomposition.

3.3. Wear Testing. After friction testing, the wear scars on the steel disk samples were examined with an optical microscope and a contactless profilometer. Optical and three-dimensional images of the wear scars for the sample lubricated with solid nanolamellar MoS_2 and WS_2, commercial MoS_2 (for comparison), and oil M8V are presented in Figure 4. With nanolamellar MoS_2 lubrication, the wear scar depth and width are less than those with nanolamellar WS_2 lubrication. The better antiwear properties of MoS_2-NL are also confirmed by the wear testing results presented in Table 1. Moreover, nanolamellar WS_2 has better antiwear properties than commercial molybdenum disulfide. Wear volumes for solid lubricants in tests above 400°C are not presented in Table 1 because of thermal deformation of the steel sample and negative wear volume.

The effect of nanolamellar tungsten and molybdenum disulfides on base oil friction and antiwear behavior was also examined. Composite oil-based lubricants were prepared

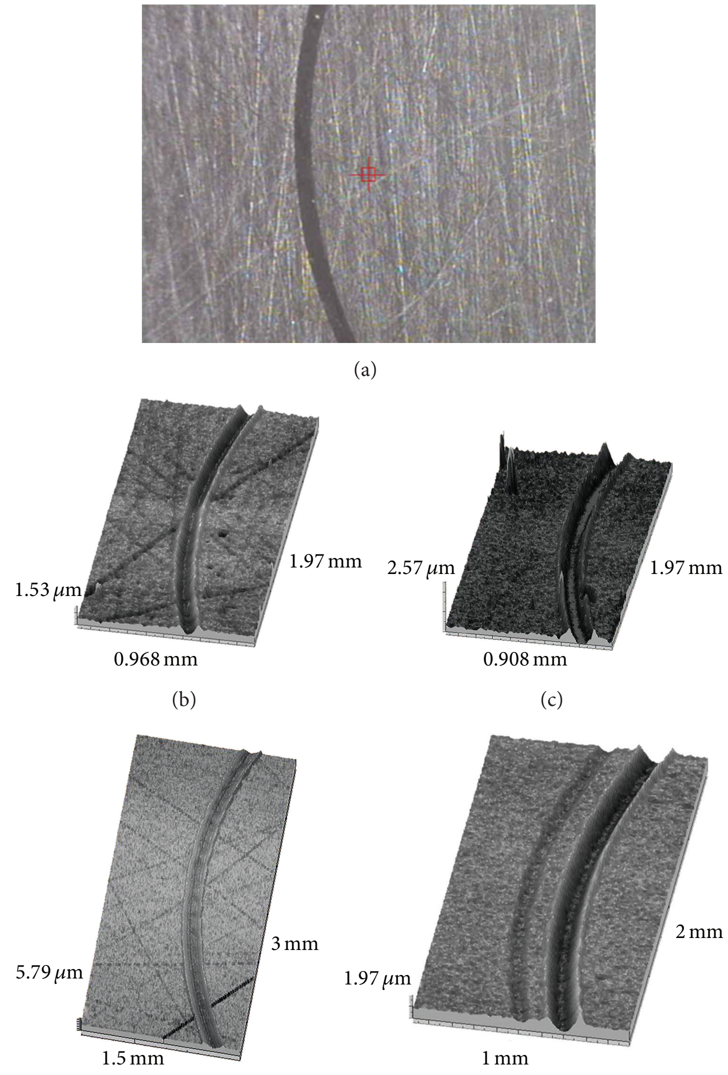

Figure 4: Optical (a) and three-dimensional images of the wear scar for the sample lubricated with solid MoS_2-NL (b), WS_2-NL (c), MoS_2-C (d), and oil M8V (e).

using 5 wt.% disulfide additives. For comparative analysis, the coefficients of friction for pure oil-based lubricants with disulfide additives were measured. In order to obtain reliable results tribological tests were carried out several times for each sample. The most representative friction test results are presented in Figure 5. The disulfide additives in oil reduce the fluctuation of the coefficient of friction but its average value changes but slightly. Apparently, this is associated with peculiarities of the method used to determine the coefficient of friction. However, the disulfide additives in oil significantly reduce the wear (Table 1).

The wear scar measurements on a contactless profilometer show that the use of nanolamellar MoS_2 in oil ensures a decrease in wear scar width from 100 to 40 μm (Figure 6) and in wear volume from $74.8 \cdot 10^3$ to $15.1 \cdot 10^3$ μm^3. As a result, the oil doped with nanolamellar disulfide displays better antiwear

Table 1: Profilometer results of wear scar.

Sample	Wear scar depth (μm)	Wear volume ($\mu m^3 \cdot 10^{-3}$)
WS_2-NL	0.48	3.1
MoS_2-NL	0.42	2.0
MoS_2-C	0.57	3.5
WS_2-NL (400°C)	2.87	NA
MoS_2-NL (400°C)	3.49	NA
Oil	0.82	74.8
Oil + 5 wt.% WS_2-NL	0.57	30.3
Oil + 5 wt.% MoS_2-NL	0.56	15.1
Oil + 5 wt.% MoS_2-C	0.75	46.7

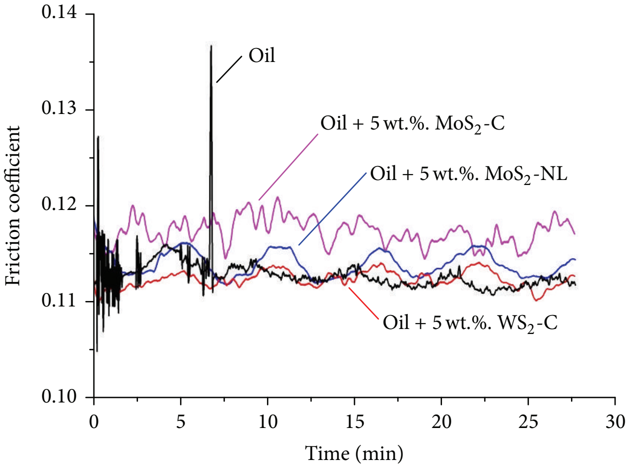

FIGURE 5: Dependence of the coefficient of friction on the presence of nanolamellar WS_2 and MoS_2 and commercial MoS_2 additives in base oil.

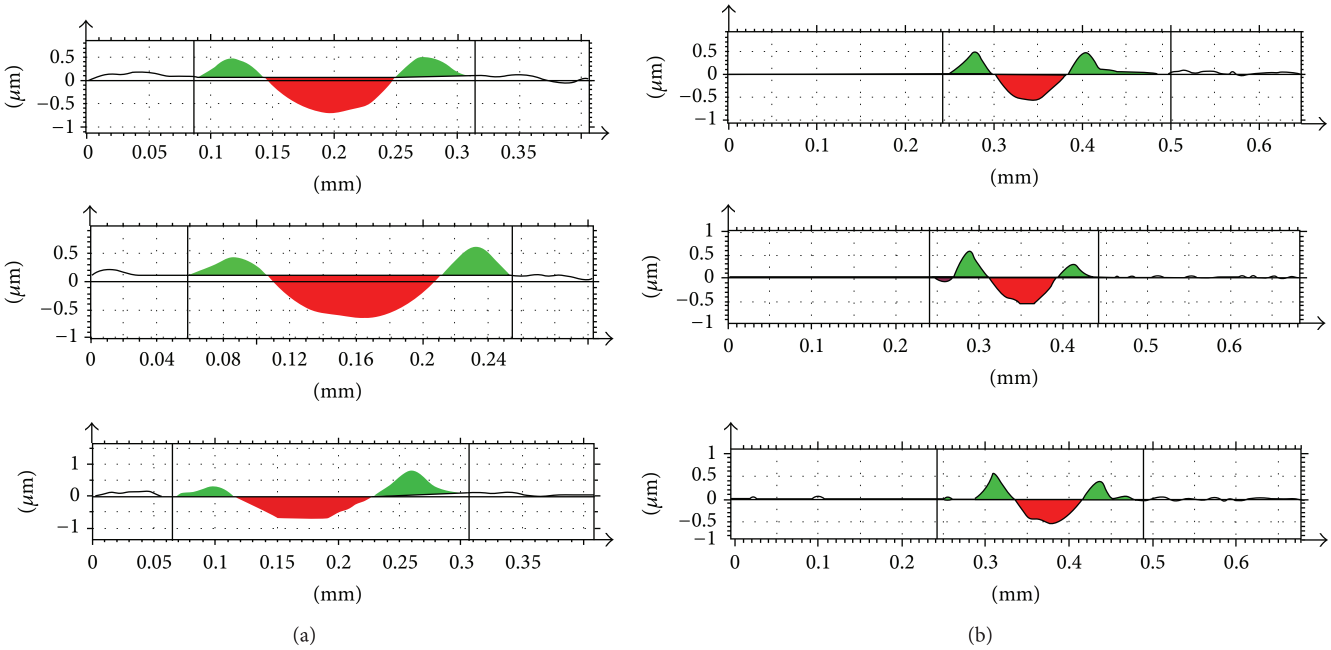

FIGURE 6: Wear scar cross-section for the samples lubricated with oil (a) and oil with 5 wt.% of nanolamellar MoS_2 (b).

performance than the base oil without nanolamellar disulfide additives.

3.4. Possible Mechanism of the Tribological Action of Nanolamellar Disulfides. Figure 7 schematically illustrates the possible mechanism of the tribological action of nanolamellar particles of tungsten and molybdenum disulfides, when they function during friction as solid lubricants or as additives to oil-based lubricants. In fact, the nanolamellar MoS_2 and WS_2 particles present blocks of hexagonal lamellae consisting of 20–40 nm thick subblocks. In its turn, the subblocks are assembled from elementary hexagonal MoS_2 and WS_2 “sandwiches” with lattice parameters c of 12.295 and 12.34 Å, respectively. Shear stress occurs during the frictional sliding process and impacts on these blocks as shown in Figure 7(1). We can assume that the van der Waals interactions between the subblocks in the nanolamellar particles are weaker than those between the elementary disulfide layers. As a result, the nanoscale subblocks start to shift from one layer to another as illustrated in Figure 7(2).

Figure 7(3) shows the final stage of the nanolamellar particle disassembling into nanoscale blocks. Finally, a nanoscale tribofilm of metal disulfide begins to form between sliding surfaces (Figure 7(4)).

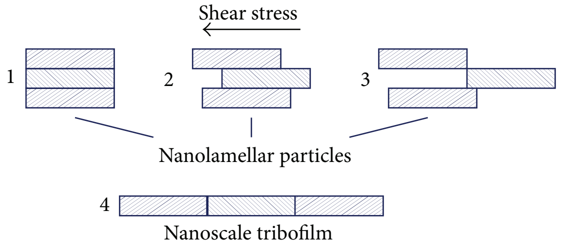

Figure 7: Schematic diagram describing the possible mechanism of the tribological action of nanolamellar disulfides: (1–3) disassembling the nanolamellar particles under shear stress, (4) formation of a nanoscale tribofilm.

The formation of a stable nanoscale disulfide tribofilm ensures lower values for the coefficient of friction and wear of sliding surfaces in comparison with those of the commercial disulfide powder. The same effect—this reduction in the coefficient of friction and wear—can be achieved by adding small amounts of nanolamellar molybdenum and tungsten disulfide to the M8V base oil. This model is to be verified with further EDS and XPS *in situ* measurements of wear tracks.

4. Conclusions

Thus, the study shows that nanolamellar MoS_2 and WS_2 synthesized by the SHS technology can reveal excellent tribological performance. This is confirmed by tribological tests of oil-based lubricants with additives of nanolamellar tungsten and molybdenum disulfide powders produced via direct reaction between nanodispersed metal powders and elementary sulfur in argon.

Analysis of the obtained results demonstrates that

(1) the friction coefficient of nanolamellar MoS_2 is 2 times lower than that of commercial powder;

(2) nanolamellar tungsten and molybdenum disulfides have low friction coefficients $\mu_{\min}$ (0.035 and 0.024) at room temperature; nanolamellar tungsten disulfide is more stable than nanolamellar molybdenum disulfide and displays a more stable friction coefficient with time at elevated temperatures (up to 400°C);

(3) the tribological tests show better antifriction behavior of oil-based lubricants with nanolamellar tungsten and molybdenum disulfide additives.

Thus, we can state that the nanolamellar tungsten and molybdenum disulfides prepared by self-propagating high-temperature synthesis demonstrate the potential as oil-based lubricant additives.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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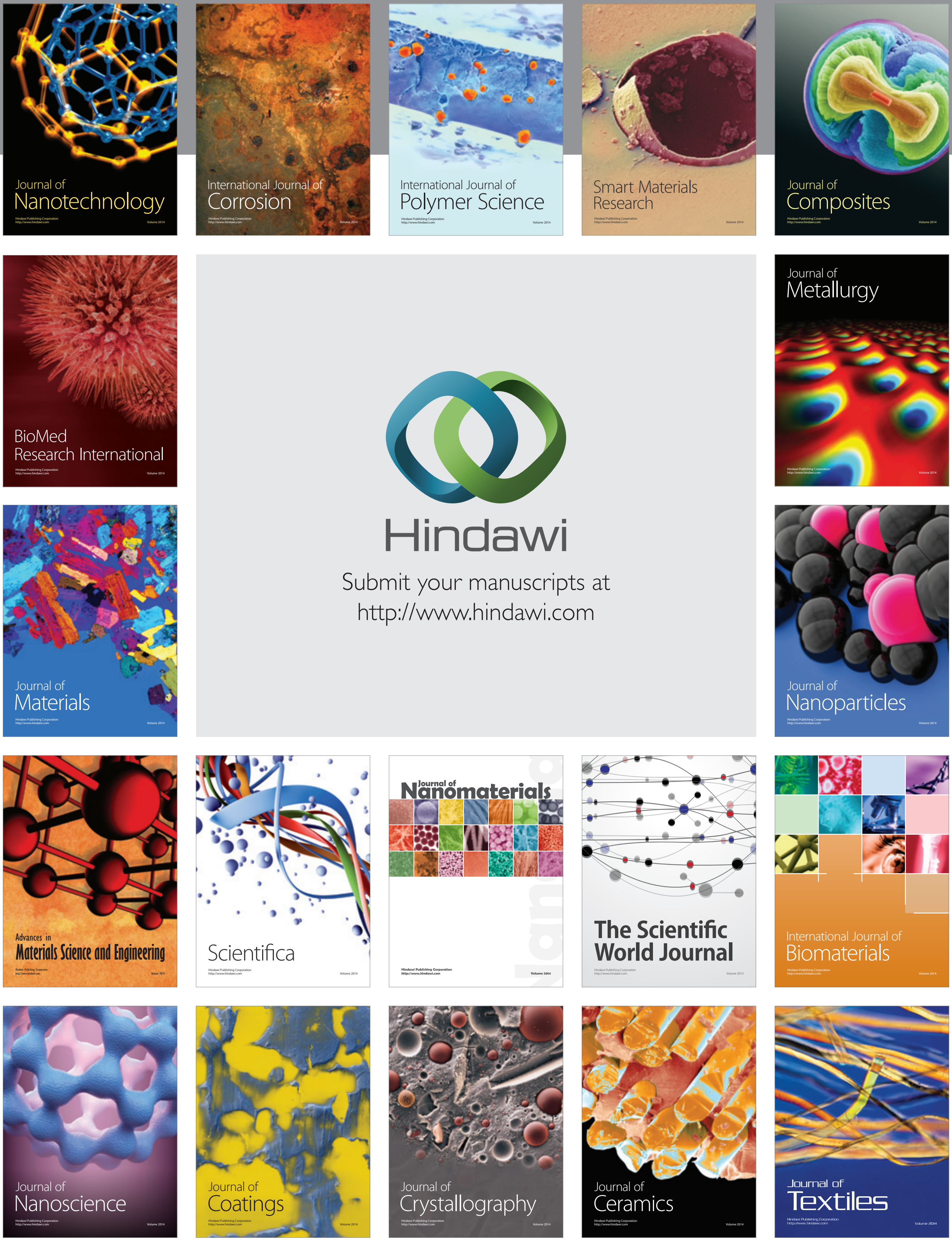
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