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A Study on the Tribological Behavior of Molybdenum Disulfide Particles as Additives

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Abstract: Molybdenum disulfide (MoS_2) is used as a solid lubricant and is well known for its tribological behavior (friction and wear). The tribological properties of the lubricating oil–MoS_2 nanoparticles mixture in different conditions of friction are studied using a four-ball tribometer, and the operating conditions of the four balls when immersed can be modeled. The current paper presents a calculating method for the critical sliding velocity (ωcr) and friction maximum torque (M_{fmax}) depending on the temperature (T) from the contact areas, obviously demonstrating low tribological performances. The film composition formed by friction, the topography, and the morphology of the particles and the friction-and-wear tracks of the balls following experiments using contact surfaces are analyzed and investigated by X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and transmission electron microscope (TEM). XPS and SEM show that nanoparticles by deposition form a protective and lubricating layer of MoS_3, which allows for an increase in the friction pair's load capacity. MoS_2 nanoparticles (n-MoS_2 of ~40 nm in diameter) compared to the common (commercial) MoS_2 particles (c-MoS_2 of ~1.5 µm in diameter) presented lower friction coefficients and higher wear-resistance values, due to the protective-layer microstructure as an intermediate lubricant between the contact surfaces. Therefore, the present paper reports the tribological properties of the lubricating oil with n-MoS_2 as an additive compared to the c-MoS_2, and by the application of the friction modeling theory using a Couette flow, it was possible to calculate the temperature, T, when the friction torque, M_f, was at its maximum, the basis on which the value of its sliding velocity, ω, was obtained corresponding to the contact areas of the four-ball system.

Keywords: MoS_2 nanoparticles; solid lubricant; XPS; SEM; TEM

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1. Introduction

MoS_2 as WS_2 and TiO_2 is a common, solid lubricant. Solid lubricants can assure the long life of friction pairs, no contamination, and can be used in environments in which liquid lubricants cannot be used.

MoS_2 in thin layers presents a low friction coefficient in dry environments, and in powder form is commonly used as an additive in oil, mixed with greases or introduced in the porous matrix of the materials. Additionally, it can be used for spray coating, pulsed laser ablation, chemical deposition, or when other lubricants cannot be used. It is well known that MoS_2 particles, especially nano-sized particles (n-MoS_2), have applications in many domains, such as solid lubrication, as additives in lubricating oils and as self-lubricating materials of type polymers [1–3]. On the other hand, n-MoS_2 in a mixture with grease, mineral oil, or powdered materials improves the tribological properties compared to typical metallic dichalcogenides particles [4–6].

To prepare ultrafine MoS_2 particles, different methods exist. However, using different methods of preparation leads to modifications of the performance and particle morphology of nano-sized MoS_2 [5–8]. Thus, for MoS_2 pure film exposed to an oxygen-rich atmosphere, the tribological properties would deteriorate as a result of oxidation. In addition, its use as a solid lubricant film can reduce the heat generated by friction [8–11].

Several types of MoS_2 nanoparticles have been experimentally investigated as possible lubrication agents, and the results suggest that this is a promising idea. Thus, MoS_2 nanotubes have been experimentally evaluated in this respect and the results are compared to reference base oil, and it can be observed that MoS_2 nanotubes significantly decrease the friction-and-wear properties compared to the base lubricant [12]. Then, in ref. [13], MoS_2 nanosheets with a thickness of about 30–70 nm were experimentally investigated by a comparison to commercial micro-MoS_2 mixed in a base oil. The results show that MoS_2 nanosheets mixed in a base oil presented better extreme-pressure values, friction reduction, and wear resistance than those with commercial micro-MoS_2. Additionally, Hu, in ref. [14], demonstrated that to obtain better tribological performances, it was necessary to provide a useful ratio for the proper mixing of nano-sized MoS_2 and commercial, common, MoS_2 particles (~1.5 μm in diameter) with different concentrations in liquid paraffin. Their lubrication capacity, friction reduction, and wear resistance were studies by measuring their extreme pressures, wear scan diameters, and friction coefficients with the help of a four-ball tribometer. The results show that the loading capacity of liquid paraffin with different kinds of MoS_2 particles increases with their contents.

Salem et al., in [2], dealt with the tribological behavior (wear rate and friction coefficient) of high-density polyethylene/MoS_2 composites against stainless steel using a linear reciprocating pin-on-disc tribometer in dry conditions. They highlighted that the use of MoS_2 enhanced the tribological performances of high-density composites compared to the polymer under dry lubricated conditions. Additionally, the scenario in relation to the MoS_2 content was discussed.

Other researchers have studied the tribological behavior of bearings' rolling material (alloy steel) in lubricated conditions with four various sizes of MoS_2 particles as additives in a conventional lubricant [15]. It was observed that as the size of the particles decreased, the friction coefficient and wear volume decreased, which was attributed to the rolling of the small-sized particles in the contact zone [15]. On the other hand, n-MoS_2 can limit the friction coefficient, especially the excessive wear, of the friction pairs sensitive to moisture [1–3,7,8,11,16].

By analyzing the mechanical properties of the materials studied and their mechanical characteristics, we can determine that MoS_2 particles mixed into a base oil manifest as an electrophoretic deposition process of particles, in general, and, in particular, MoS_2 particles. This process can be reported kinetically via the use of response surface methodology based on kinetic models, using both statistical and quantitative techniques [17,18]. The fundamentals of electrophoretic-deposition-process kinetics are useful to demonstrate the influence of process parameters on the properties of deposited films in an interval of time. Therefore, the kinetics of the electrophoretic deposition process was initiated as a function of tension and time of deposition, to avoid a significant loss of MoS_2 particles, which decreases during the process, followed by deposited films' physicochemical characterizations (XPS and SEM).

According to ref. [17,18], the kinetic models can reproduce experimental data well, alongside some other semi-empirical equations of kinetics, which help us to comprehend the process of thermal degradation in depth; this is a useful tool in anticipating thermal-stability properties, avoiding the thermal degradation of MoS_2 products for industrial applications.

Thus, the analysis of solid lubricant films demonstrates, on the four-ball tribometer, the utility of lubricating sliding and rolling dry contacts for long time periods [7,8,14,19]. Further studies [7,8,11,20] show that, regarding the balls, reservoirs developed in the terminal points of wear tracks or around the perimeter of the wear scars, where the lubricant could be stored. The conditions of friction of the four balls in the lubricating mixture (mineral oil with MoS_2 particles) can be modeled with the help of the Couette flow, which allows for the calculation of the temperature (T) when the friction torque (M_f)

is maximal. Additionally, the sliding velocity (ω) corresponding to the maximal friction torque (M_{fmax}) and its value can be determined [21–23].

Therefore, this study presents the tribological properties of a lubricating oil with n-MoS_2 as an additive compared to c-MoS_2. Moreover, the friction modeling theory is applied using the Couette flow for temperature calculation when the friction torque, M_f, is at its maximum level. On this basis is obtained and the value of the sliding velocity, ω.

2. Materials and Methods

In the experiments, n-MoS_2 (~40 nm in diameter) and c-MoS_2 (~1.5 μm in diameter) were mixed in a base oil (engine oil 15W/40 Super 2) and compared to assess their tribological performances. These dimensions were chosen for n-MoS_2, being of a medium size and obtained through a simple method of grinding and selection by sieving, and c-MoS_2 is commercially available. The mixing of n-MoS2 and c-MoS2 in base oil 15W/40 Super 2 led to tribological results that were different from the results previously obtained for other nanoparticles.

For this, the n-MoS_2 and c-MoS_2 particles were dispersed in the base oil by means of ultrasonic methods in order to study their lubrication capacity, friction reduction, and wear resistance. Simultaneously, the mixture of lubricating oil and n-MoS_2 or c-MoS_2 was homogenized by the quick homogeneous precipitation method (QHPM) and the gas-solid reaction of MoO_3 (molybdenum trioxide) obtained from Na_2MoO_4 (sodium molybdate) and Na_2S (sodium sulfide) at an ambient temperature.

The tribological performances (extreme load, friction behavior, and wear resistance) were tested and measured on a four-ball tribometer (Rtec-Instruments, Yverdon-les-Bains, Switzerland), where steel balls, RUL 2, with 60–62 HRC hardness and a diameter of 12.7 mm were used.

The physical and chemical properties of the materials used in the experiments are shown in Table 1.

Table 1. Materials used with physical and chemical properties.

Materials	Physical and Chemical Properties
Molybdenum disulfide MoS_2	Molar mass:160.07 g/mol; appearance: black/lead-gray solid; density: 5.06 g/cm^3; melting point: 2.375 °C; solubility in water: insoluble; solubility: decomposed by aqua regia, hot sulfuric acid, nitric acid, insoluble in dilute acids; band gap: 1.23 eV (indirect) and ~1.8 eV (direct, monolayer)
Engine oil 15W/40 Super 2	Molecular weight: no data; color: yellow-brown to black; physical state: liquid, oily; melting point: −34.4 °C; boiling point: 360.0 °C; viscosity at 25 °C: variable (104 cSt at 40 °C and 14.9 cSt at 100 °C); odor: lube oil odor; odor threshold: no data; solubility: insoluble in water at 20 °C and no data for organic solvent(s); vapor pressure at 20 °C: no data; Henry's law constant: no data; auto-ignition temperature: $\geq$ 135 °C; flashpoint: $\geq$ 163 °C; flammability limits: no data; conversion factors: no data; explosive limits: no data
Steel RUL2	Thermal treatment: re-bake at 720–800 °C, tempered at 830–870 °C in oil, returned to 150–300 °C; hardness: 60–62 HRC; tear resistance: 1150–1800 MPa; flow resistance: 920–1440 MPa; normal modulus of elasticity: $1.95{\cdot}10^5$–$2.1{\cdot}10^5$ MPa, transverse modulus of elasticity: $0.78{\cdot}10^5$–$0.815{\cdot}10^5$ MPa; density: 7850 kg/m^3; melting point: 1510 °C; Poisson's ratio: 0.27–030; thermal expansion (10^{-6}/K): 9.0–15.0; thermal conductivity (W/m-K): 26.0–48.6; specific heat (J/kg-K): 452–1499; electrical resistivity (10^{-9} W-m): 210–1251; chemical composition: 0.95%–1.1% C, 1.3%–1.65% Cr, 0.25%–0.45% Mn, 0.15%–0.35% Si, max. 0.030% P; max. 0.030% S

In addition, the viscosity variation with temperature for the lubricant mixture (base oil + n-MoS_2) was represented graphically because viscosity represents one of the most important properties of lubricants, which decides their behavior. The viscosity at different temperatures was determined using a capillary viscometer of the Ubbelohde type (Meck KGaA, Darmstadt, Germany), which expresses the Poiseuille law:

$$V = \pi r^4 \Delta pt/8l\eta \quad (1)$$

where V is the lubricant-mixture volume with dynamic viscosity η, which flows at a certain time t, through a capillary length l and radius r, under the pressure difference Δp.

The relation (1) results in the dynamic viscosity $\eta = \pi r^4 \Delta pt/8lV$, whose temperature variation is presented in Figure 1, and it can be observed that it is similar to that of a Newtonian fluid, so the lubricant mixture (base oil + n-MoS_2) behaves similar to a Newtonian fluid.

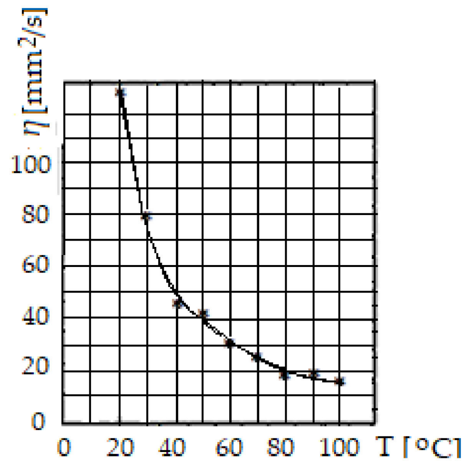


Figure 1. Curve of viscosity–temperature variation for the lubricant-mixture base oil + n-MoS_2 (MoS_2 nanoparticles).

The morphology of the particles was observed with a transmission electron microscope (Bruker Corporation, Karlsruhe, Germany) (TEM with beam energy in the range of 100,000 to 400,000 eV, capable of providing very-high-resolution images down to a level of several Angstroms (~0.19 nm), providing high brightness levels and a high-stability-electron source for use at 100 and 200 kV), the topographies of the contact surfaces were scanned with SEM (Tescan Oskay Holding, Brno-Kohoutovice, Czech Republic) (100,000× amplification power, secondary-electron detector, advanced vacuum, tungsten filament, carousel 7 samples (standard dimensions 10 × 10 × 45 mm^3), software and 2D and 3D structural surface analyses) and the friction surface's chemical state was analyzed with XPS (using a pass energy of 188 eV and Mg Ka line-excitation source with the reference C1s at 284.6 eV) to remark on the surface's modification under the influence of n-MoS_2 particles.

3. Theoretical Considerations

Using the Couette flow, the friction between the four balls of the ball tribometer could be modeled. For this, it was assumed that lubricant-mixture cooling occurred only by conduction. The friction torque (M_f) and power lost by friction are expressed by the following equations:

$$M_f = C\eta\omega \quad (2)$$

and

$$C\eta\omega^2 = AK\omega^{\alpha}(T - T_0), \quad (3)$$

where C–parameter that takes into account the change in viscosity due to movement; η—dynamic viscosity of the lubricant; ω—angular velocity; $A = \pi r_f^2$—friction area with r_f-friction radius; K—heat-transfer coefficient; α—dependent constant on cooling conditions; T—operating temperature and T_0—initial temperature.

By replacing ω from Equation (2) with (3), we obtained the relation of the torque–temperature variation:

$$M_f = [AK(C\eta)^{1\text{-}\alpha}(T - T_0)]^{1/(2\text{-}\alpha)}, \tag{4}$$

Any viscosity–temperature variation law can be considered here.

The derivative of friction torque via temperature is:

$$\frac{dM_f}{dT} = \left[\frac{AKC^{1-\alpha}}{\eta(T - T_0)^{1-\alpha}}\right]^{1/(2-\alpha)} \cdot \frac{1}{2-\alpha}\left[(1-\alpha)(T - T_0)\frac{d\eta}{dT} + \eta\right] \tag{5}$$

From the condition as derivative of M_f with T set as zero is obtained the following equation:

$$(1-\alpha)(T - T_0)\frac{d\eta}{dT} + \eta = 0 \tag{6}$$

The solution of this equation allowed us to obtain the temperature T_{cr} at which the M_f is maximum.

Considering the usual viscosity–temperature variation law, $\eta = \eta_0 \; exp[-\beta(T - T_0)]$, the expressions of the critical temperature, T_{cr}, where η_0 is the dynamic viscosity of the lubricant at T_0 are presented in Table 2, and β is the exponent that takes into account the viscosity-concentration dependence.

Table 2. Expressions of T_{cr} at which M_f is at its maximum level.

η(T)	**T_{cr} in the Situations:**			
	In general	In different cooling conditions		
Case		$\alpha = 0^a$	$\alpha = 1/3^b$	$\alpha = 1/2^b$
$\eta = \eta_0 \; exp[-\beta(T - T_0)]$	$T_0 + 1/\beta(1 - \alpha)$	$T_0 + 1/\beta$	$T_0 + 1.5/\beta$	$T_0 + 2/\beta$

Note: [a] The cooling is independent of velocity, ω. [b] The cooling is proportional to velocity, ω.

The results of the friction maximum torque M_{fmax} and corresponding velocity ω (similar to Equations (2) and (4)) are expressed as:

$$M_{fmax} = [AK(C\eta_{cr})^{1\text{-}\alpha}(T_{cr} - T_0)]^{1/(2\text{-}\alpha)} \tag{7}$$

and

$$\omega_{cr} = M_{fmax}/C\eta_{cr}, \tag{8}$$

where η_{cr}–the critical dynamic viscosity evaluated at temperature T_{cr} from the law used for the variation of viscosity with temperature, and presented in Table 2.

4. Results and Discussions

The n-MoS_2 particles as additives in different concentrations were investigated from a tribological point of view on the four-ball tribometer, in comparison to c-MoS_2 particles mixed in mineral oil (15 W/40 Super 2–base oil). Thus, Figure 2 can be observed as an evolution of extreme load depending on the concentration and particle size of MoS_2 in the base mineral oil.

The experimental results obtained for the four-ball tribometer present the extreme load of lubricating oil (base oil + n-MoS_2) with a particle content greater than or equal to 0.5 wt. %, which is higher, by about 30%, than the same oil with the same c-MoS_2 particle content (see Figure 2). Additionally, the experimental results show that the loading capacity of the base oil with n-MoS_2 and c-MoS_2 particles increases with the decrease in their size.

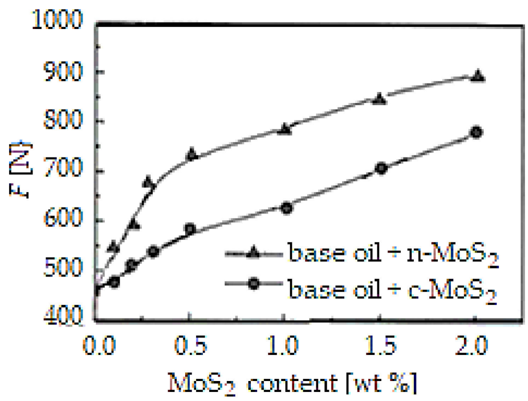


Figure 2. Variation of extreme load depending on the MoS_2 particle's content via the four-ball tribometer (n-MoS_2 = MoS_2 nanoparticles; c-MoS_2 = common MoS_2 particles).

The friction coefficient variation with the time, t, and angular sliding speed, ω, can be observed in Figure 3a and b as an experiment used to monitor its variation (of the friction coefficient). Figure 3 shows that the friction coefficient's variation vs. time and angular sliding speed for all the three cases (base oil, base oil + c-MoS_2, base oil + n-MoS_2) is relatively small, with closed shapes, which indicate that their friction process and chemical reaction are comparatively simple, no matter the parameter (t or ω) through which their variation is presented.

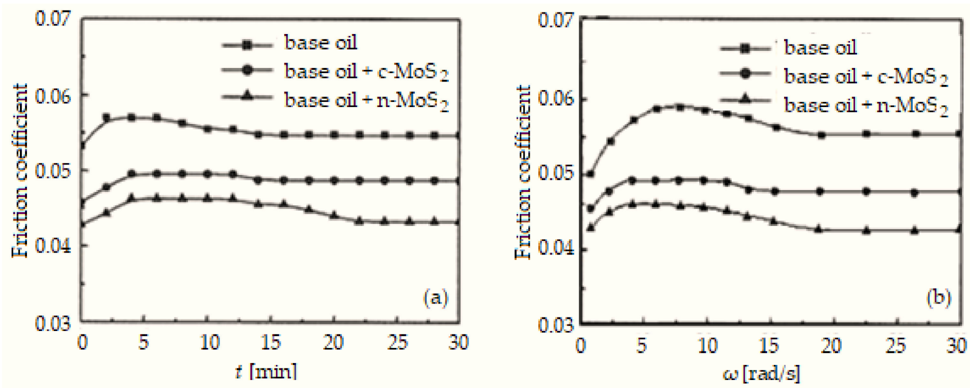


Figure 3. Friction coefficient variations depending on time, t (**a**) and angular sliding speed, ω (**b**) on a four-ball tribometer.

However, it can be observed that the base oil containing c-MoS_2 and n-MoS_2 particles had friction coefficients smaller than the base oil. Although the friction coefficient of the friction pairs with base oil + n-MoS_2 was the smallest, there was a discrete difference compared to that of the base oil + c-MoS_2. Thus, it can be said that the base oil + n-MoS_2 presented a more predominant anti-friction performance than the base oil + c- MoS_2. In addition, the base oil + n-MoS_2 had the lowest friction coefficient, and as a result it had greater chemical activity than the base oil + c-MoS_2, especially the simple base oil.

Additionally, for the whole friction process it was observed that the friction coefficients for all cases increased relatively slightly after 5–10 min, and after 10–15 min they decreased relatively slightly. The prolonged time enabled heat energy to accumulate and permitted further tribochemical reactions between the active base oil + n-MoS2 and friction-pair materials.

Therefore, the base oil + n-MoS_2 had a better extreme load, friction-reducing performance, and better wear resistance than the base oil + c-MoS_2, especially for the pure base oil.

Figure 4 shows the M_f variation depending on ω. This variation included the experimental results for the four-ball tribometer of the lubricant mixture (oil additive with MoS_2 particles). We observed a non-linear variation of M_f in the ratio with ω and, in particular, its decrease, over a critical velocity, ω_{cr}, which was due to the viscosity variation of the lubricant mixture with the temperature.

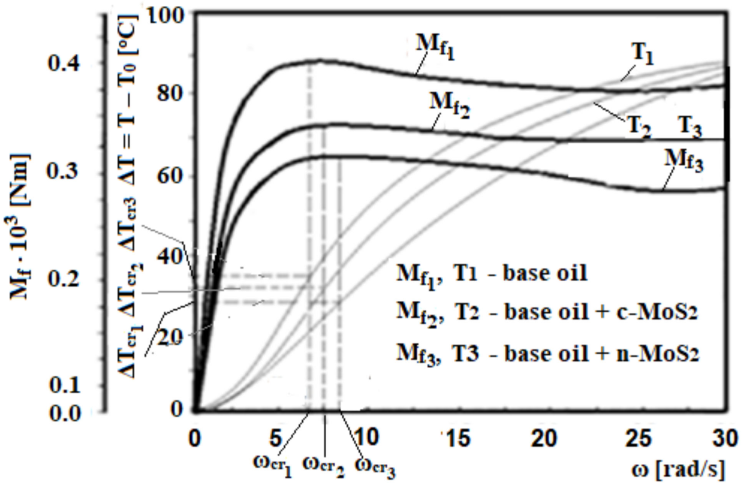


Figure 4. Variations of M_f and T depending on ω (results obtained experimentally for base oil, base oil + c-MoS_2, and base oil + n-MoS_2).

The non-linear variation of M_f in the ratio with ω was explained by the fact that M_f increased with ω, until it reached a maximum value for each of the three situations analyzed (base oil, base oil + c-MoS_2, and base oil +n-MoS_2), and ω corresponding to these values was considered as the critical speed (ω_{cr}) and the corresponding temperatures were critical temperatures (T_{cr}). This was possible because the lubricant mixture adhered to the contact surfaces; its temperature slightly increased and was sufficiently viscous for M_f to increase to a maximum value (when $\omega = \omega_{cr}$), after which it followed a relatively slow decrease when the temperature, T, continued to increase with the increase in ω and the viscosity of the lubricant decreased.

The theoretical model used and presented in ref. [21–24] describes the tangential stress variation with velocity for the Couette flow. This model illustrates the shape of the $M_f(\omega)$ variation presented in Figure 4, but does not predict the functions of M_{fmax}, ω_{cr}, and T_{cr} explicitly. The method for the calculations of $M_f(T)$ and $\omega_{cr}(T)$ is shown in the theoretical considerations above.

The experimental results presented in Figure 4 are for the 15 W/40 Super 2 mineral oil (the base oil), base oil + c-MoS_2 (additive with c+MoS_2 by the 1.5 µm size), and base oil + n-MoS_2 (additive with n-MoS_2 of 40 nm size).

From the viscosity–temperature curve of the lubricating mixture (see Figure 1), the result is $\beta = 0.0266$–0.0436 for the considered temperature interval. In addition, because ω decreased, it could be estimated that $\alpha = 0$, and thus $\Delta T_{cr} = T_{cr} - T_{\_0} = 1/\beta = 28, \ldots, 35$ °C.

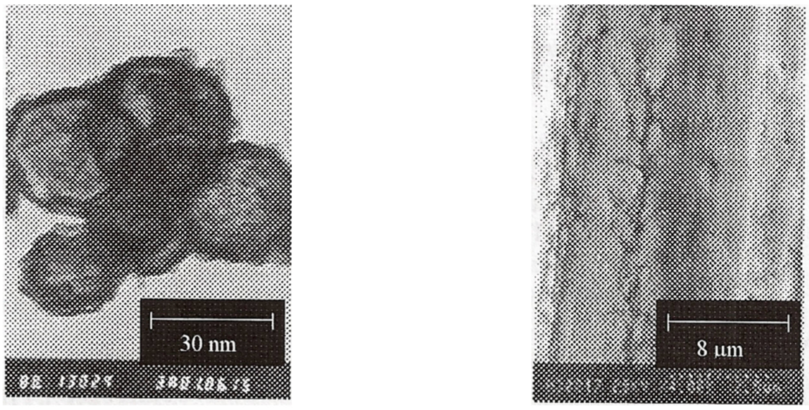
The chemical state and composition of the elements of the rubbed and worn balls' surfaces were analyzed on XPS, and on SEM, the topographies of the same surfaces lubricated under different conditions were observed.

The improvements regarding the extreme load and friction reduction in the friction pairs from the four-ball tribometer could be explained by the easier adsorption of the n-MoS_2 on the balls' sliding surfaces and the formation of a protective and lubricating film containing an oxide, namely, MoO_3 (molybdenum trioxide). The MoO_3 oxide was obtained from this film after the rapid oxidation of nanoparticles in the sliding process, which were also maintained by the release and furnishing of the nanoparticles from the topography valleys onto the rubbing metal surfaces and their confinement at the interfaces. Simultaneously, the analyses of the surface film composition, characterized with the help of XPS, and of the SEM images showed that the deposed nanoparticles formed a protective film (MoO_3) allowing for an increase in the load capacity of the friction pairs.

In addition, by XPS and SEM analyses, it turned out that the difference in the tribological performance between n-MoS_2 and c-MoS_2 particles was due to the surface effect and interfacial-size effect of nanoparticles and the formation of MoO_3 thin film on the rubbed surface.

SEM analysis showed that the worn surfaces were smooth when using n-MoS_2 as an additive, and XPS analysis showed that more MoO_3 existed on the rubbed contact surfaces and FeS in the anti-friction and anti-wear thin films, compared to using c-MoS_2.

The n-MoS_2 morphology observed using TEM and the topographies of contact surfaces of the upper ball in the four-ball experiment, analyzed with SEM, is shown in Figure 5.



(**a**) (**b**)

Figure 5. Images of n-MoS_2 morphology by TEM (**a**) and of the contact surface by SEM (**b**).

5. Conclusions

- Adding n-MoS_2 particles to the lubrication oil improved the tribology performances (of the extreme load, the friction, and wear resistance). The good tribological properties of n-MoS_2 were mainly ascribed to the surface effect of nanoparticles.
- The influence of the n-MoS_2 particles on the friction coefficient and extreme load of the lubricating mixture (base oil + n-MoS_2) may have been due to the easier adsorption of n-MoS_2 particles on the ball's sliding surfaces and the formation of an oxide MoO_3, which is a protective and lubricating film.
- This was possible because of the relatively easy oxidation of n-MoS_2 in the sliding process, following its release and traveling from the valley onto the contact metallic surfaces and their isolation at the interface. Moreover, the formation of the MoO_3 complex film and FeS on the rubbed surface also played an important role in friction reduction and wear resistance.
- Using XPS and SEM, it was suggested that the difference in the tribological performance between n-MoS_2 and c-MoS_2 particles was attributed to the surface effect and interfacial-size effect of nanoparticles and the formation of MoO_3 thin film on the rubbed surface.
- Experimental results obtained via the four-ball tribometer show that with an n-MoS_2 content greater than or equal to 0.5 wt. %, the extreme load of additive lubricating oil was greater by about 30% than the same additive lubricating oil with c-MoS_2 with the same percentage content. At the same time, the results show that the loading capacity of base oil with different kinds of MoS_2 (here, n-MoS_2 and c-MoS_2) particles increases with the decrease in their size.
- The lubricating oil mixed with n-MoS_2 could obviously decrease the friction coefficient, and the results show that the size of the MoS_2 nanoparticles influences, to some extent, the value of the friction coefficient.
- The base oil containing the mixture of n-MoS_2 particles had a better extreme load, friction-reducing performance, and better wear resistance than the base oil containing c-MoS_2 particles or pure base oil.

- Additionally, β = 0.0266–0.0436 and ΔT_{cr} = 28, . . . , 35 °C values were obtained from the diagram of the viscosity–temperature variation curve and the M_f and T variations with ω, respectively, which demonstrates that the calculated results are in accordance with the experimental ones.
- The methods for determining T_{cr} were simple and could be used to choose both the working T and the size of the n-MoS_2 particles as additives in the base oil. Additionally, M_{fmax} could be established for the friction pairs of the machine elements that were required and for the friction pairs of the machine elements where friction was undesirable.
- Therefore, the friction and wear phenomenon of n-MoS_2 in the lubricating oil are still not clearly defined, so they are worth studying in the future, including the relationship between the properties and the tribological results of n-MoS_2 at the micro-level. It is also worth studying the mixing effect of c-MoS_2 and n-MoS_2 in the actual lubricating oil with various additives.

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