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MATERIALS
ENGINEERING

Tribological synergy between classical ZDDP and innovative MoS_2 and MoO_3 nanotube additives at elevated temperatures

Agnieszka Maria Tomala[a,b*], Manel Rodríguez Ripoll[b], and Remigiusz Michalczewski[a]

[a] ITeE-PIB Institute for Sustainable Technologies – National Research Institute, 26-600 Radom, Poland
[b] AC2T research GmbH, 2700 Wiener Neustadt, Austria

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Abstract. One of the most important drawbacks limiting the application of MoS_2 nanotubes (NTs) as an oil additive is their temperature sensitivity. Recent studies showed that MoS_2 NTs can be substituted by MoO_3 NTs in conjunction with S-containing lubricants by exploiting a novel approach of in-situ tribochemical sulphurization. The objective of this work was to investigate the temperature influence on tribological properties of innovative lubricant additives in the form of MoS_2 and MoO_3 NTs. The NTs were mixed in base oil with and without the presence of S-containing additives. The tribological performance was investigated using a SRV reciprocating sliding testing machine in a steel ball on a steel disc configuration under temperature ramping conditions. The results showed very positive synergy between the traditional anti-wear additive and the innovative MoO_3 and MoS_2 NTs, causing superb tribological performance up to temperatures of 200 °C. The presented findings show that the in-situ sulphurization of MoO_3 NTs was promoted by using the traditional zinc dialkyl dithiophosphates (ZDDP) anti-wear additive, which ensured the stability of this additive combination at severe oil temperature and tribotest conditions. The tribochemically formed tribofilm derived from ZDDP and in-situ sulphurized MoO_3 was much thicker compared to other lubricating blends investigated in our research so far.

Key words: MoS_2 nanotubes, MoO_3 nanotubes, temperature ramp, additives, tribofilm, friction.

1. INTRODUCTION

Transition metal dichalcogenides (TMDs) were one of the first layered compounds found to be suitable for friction and wear reduction. Due to their 2D hexagonal arrangement, some TMDs such as MoS_2 have a transition metal atom sandwiched between two chalcogen atoms forming a S–Mo–S layer [1].

Initially TMDs were applied as burnished or sputtered coatings in dry friction regime [2]; however, after the pioneering synthesis of TMDs as inorganic fullerene-like nanoparticles [3], it became possible to make use of their potential as a lubricant additive. Shortly after, a number of researchers showed the great tribological potential of nanolubricants containing TMD nanoparticles and other morphologies including nanotubes [4–6]. The tribological performance of TMD particles is strongly affected by their morphology, structure, and size along with the large influence of the test conditions [7]. So far the main conclusion has been that MoS_2 in the form of multiwall nanotubes (NTs) contains more defects than the inorganic fullerine–molybdenum disulphide (IF-MoS_2) in the form of platelets, and therefore it is easily exfoliated [4]. One of the main advantages of establishing TMD nanoparticles as a friction modifier in commercial fully formulated products lies in their ready availability in large quantities.

* Corresponding author, agnieszka.tomala@itee.radom.pl

The synthesis of TMD nanoparticles relies on the sulphurization of inorganic compounds of transition metals, such as MO_3, MCl_5, MF_6, $Mo_6S_xI_y$, WO_x–$W_{18}O_{49}$, or transition metal–organic precursors, such as $M(S\text{-}t\text{-}Bu)_4$ and $M(CO)_6$ at high temperatures, mostly in the presence of H_2S or a similar sulphur compound [8,9]. Consequently, an in-situ sulphurization of TM-based precursor nanoparticles during operation would provide an economical option for skipping the sulphurization step, thus improving the efficiency of the process by reducing the costs and saving energy. Sulphurization requires high temperature and pressure; both these conditions are typically found between sliding bodies at the interacting asperities [10]. This approach assumes the presence of a transition metal oxide in conjunction with a sulphur compound in the lubricant formulation [11]. This condition is nowadays already fulfilled by most of the commercially available fully formulated lubricants, since they contain anti-wear (AW) additives such as zinc dialkyl dithiophosphates (ZDDPs) or extreme pressure additives, such as sulphurized olefins [12].

The aim of this work was to investigate the influence of temperature on nanolubricants containing MoS_2 and MoO_3 NTs in combination with the traditional ZDDP AW additive, promoting the in-situ sulphurization referring to [11]. The results show a very positive influence of the AW additives of both types of NTs on the friction and wear of tribopairs. However, the temperature limiting the effectiveness of MoS_2 nanotubes accompanied by an AW additive turned out to be lower compared to MoO_3 accompanied by an AW additive. This suggests that the sulphurization process improves the tribological performance of nanoparticles-based additives at elevated temperatures.

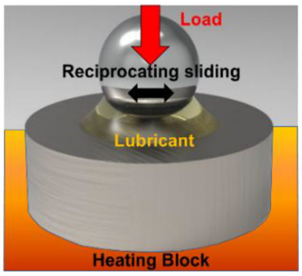
2. MATERIALS AND METHODS

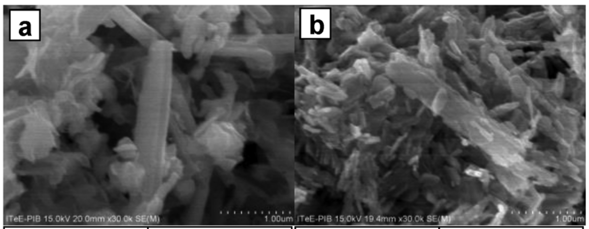
The tribological tests were performed on a SRV® tribometer (Optimol Instruments Prüftechnik GmbH, Germany) under reciprocating sliding conditions using a point contact with the test parameters given in Table 1. The material used in tribological tests was bearing steel 100Cr6 (1.3505) with a microstructure formed by fine martensite and disperse micrometre-size carbides, which resulted in a hardness of 850 HV10 with a roughness of 0.05 µm. As a counterbody, the same bearing steel balls with the same hardness and roughness and a diameter of 10 mm were used.

The base oil used in this study was NEXBASE® 2008 polyalphaolefine 8 (PAO) with a viscosity of 8 mm^2/s at 100 °C. The AW additive used was a 99% pure mixed primary/secondary ZDDP from Afton Chemicals Corporation. The MoS_2 and MoO_3 NTs investigated in this study were synthesized from $Mo_6S_2I_8$ nanowires by the procedure reported in [13,14]. The diameter of the NTs was in the range of 100–150 nm, while their length was up to 3 µm as illustrated in Fig. 1a (MoS_2) and in Fig. 1b (MoO_3). The walls of the NTs were approximately 10 nm thick and formed dome terminations. The lubricant mixtures containing NTs were homogenized using an ultrasonic processor UP200H (Hielscher – Ultrasound Technology). The selected sonication parameters at the probe tip for 10 mL lubricant blend were 20% amplitude during 5 min, while the pulse was on and off for 2 s, respectively. The AW additives with MoS_2 NTs and MoO_3 NTs were blended with PAO oil in 2% weight concentration.

After the tests the tribologically investigated samples were rinsed with petroleum ether followed by cleaning in an ultrasonic bath by isopropanol for 3 min (both solvents were HPLC grade). Hereafter, the wear tracks were examined by using a series of surface characterization methods including the use of an optical microscope, optical interferometer, and scanning electron microscopy (SEM) with energy dispersive X-ray spectroscope (EDS).

Table 1. Summary of SRV reciprocating sliding tribotest parameters including a schematic of the experimental set-up

Tribological test set-up	***SRV® test*** 
Contact conditions	Point contact at reciprocating sliding
Disc – Ball material	Steel – steel
Track length	1 mm
Speed	0.1 m/s (50 Hz)
Normal load	100 N
Mean contact pressure	1.46 GPa
Test duration	60 min
Temperature	40–220 °C
Lubricant blends	Reference polyalphaolefine (PAO) PAO + 2 wt% MoS_2 NTs PAO + 2 wt% MoO_3 NTs PAO + 2 wt% AW + 2 wt% MoO_3 NTs PAO + 2 wt% AW + 2 wt% MoS_2 NTs
Measured parameters	Coefficient of friction vs time at ramping temperature, wear volume on discs, and wear scar diameter on balls



Element	Atom%
C	17.39
O	36.02
S	27.13
Mo	19.45

Element	Atom%
C	9.90
O	74.88
Mo	15.22

Fig. 1. SEM micrograph and EDS analyses of the (a) MoS_2 and (b) MoO_3 nanotubes powder.

Optical measurements of the balls wear scar diameter with the precision up to 1 μm were realized using Nikon MM-40/L3FA. The surface topography of the tested samples was evaluated by using a Taylor Hobson CCI HD non-contact 3D optical profiler. Surface roughness was measured before and after the tests according to ISO 4287. The TalyMap Platinum (v. 6.2.7487) software was used for wear volume analysis.

The SEM micrographs were obtained with Hitachi SU-70 analytical field emission SEM. It is equipped with the Schottky electron source with ultra-high resolution and EDS (from Thermo Scientific).

3. RESULTS

3.1. Friction characteristics

In order to understand the influence of temperature on the friction properties of the lubricants containing MoS_2 and MoO_3 nanotubes, reciprocating sliding tests with ramping temperature were carried out. The SRV results are presented in the form of friction scans as a function of time, while after 10 min stabilization at 40 °C the temperature was rising 3.6 °C/min up to 220 °C (Fig. 2a). It is important to note that the error bars presented on the friction curves represent the average of three repetitions, and it can be stated that the reproducibility of the results was very high, particularly during the first 10 min when the temperature was stable and kept constant at 40 °C; in the later stage when the temperature gradually increased the reproducibility was slightly lower (Fig. 2a). The use of PAO oil results in a very high coefficient of friction (CoF) values at the initial brief running (first minute of the tests), which is not observed for the lubricants containing additives (Fig. 2a). This initial stage of unstable running has a vast effect on the error bar for PAO in Fig. 2b, as well as high wear rate values presented later in this manuscript.

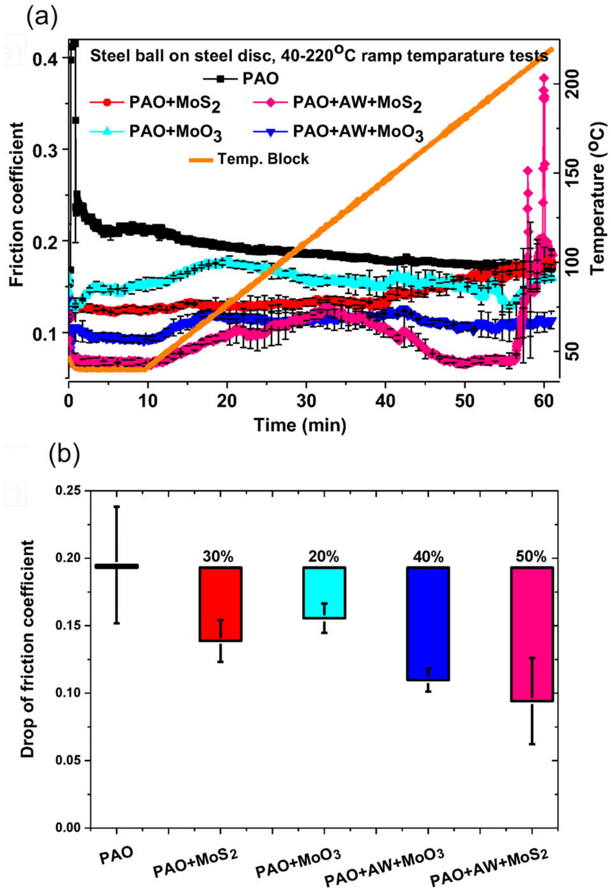


Fig. 2. Scan of CoF over the test duration under ramping temperature (a) and determinative mean values of CoF and standard deviation for the entire friction scan (b) for the studied lubricating blends: reference base oil PAO, PAO mixture containing 2 wt% MoS_2 nanotubes, PAO mixture containing 2 wt% MoO_3 nanotubes, PAO blended with 2 wt% AW additive and 2 wt% MoO_3 NTs, PAO blended with 2 wt% AW additive and 2 wt% MoS_2 NTs.

Figure 2b shows the reduction of the GoF for four lubricating blends containing MoS_2 and MoO_3 NTs compared to the reference base oil PAO. As expected and reported previously [5], a strong friction reduction (by over 30% compared to the base oil PAO) can be observed for the lubricant blend containing MoS_2 NTs in the steel–steel material configuration (Fig. 2b). The results indicate that the mixture containing MoO_3 NTs alone is not so effective in friction reduction (less than 20%) as the MoO_3 NTs accompanied with an AW additive (PAO + AW + MoO_3), where the friction reduction was 40%.

The lowest friction values were observed for MoS_2 NTs accompanied with AW additives; there the initial friction level was 0.07 (PAO + AW + MoS_2 in Fig. 2a). However, this lubricating blend turned out to be the most unstable under increasing temperature; the temperature completely liming its effectiveness was about 200 °C. The last 5 min of the unstable friction scan for the blend PAO + AW + MoS_2 (Fig. 2a) affected the error bar (Fig. 2b), and also caused high wear rate values presented later in this article.

3.2. Wear results

The volume analysis method estimates the volume of a worn material based on the measurement of the wear scar bottom and the top of the surface reference plane. The reference plane was set as the average height of the unworn area outside the wear track. The wear volume was calculated for the whole wear track, and only the area under the reference plane was considered. In addition, wear tracks on the balls and discs were visually inspected using an optical microscope.

Figure 3 illustrates wear results under ramping temperature with five different lubricants. The highest wear on the steel disc occurred after the SRV tests with PAO + MoO_3, and the highest wear on the ball after the test with pure base oil PAO. Surprisingly, the wear was relatively high for PAO + MoS_2, indicating that MoS_2 alone is very sensitive to high temperature.

To recap Fig. 2 and Fig. 3, the presence of MoS_2 and MoO_3 NTs lowered the friction coefficient in all measured temperature ranges; nevertheless, these NTs did not provide comprehensive surface protection. In order to achieve both benefits simultaneously, it is necessary to combine classical AW additives and innovative NTs.

As expected, the lowest wear volume on the discs was observed when an AW additive was present in the lubricating blend. This is not surprising as ZDDPs are known to provide wear protection to steel substrates by forming a soft tribofilm containing Zn, S, and P that is

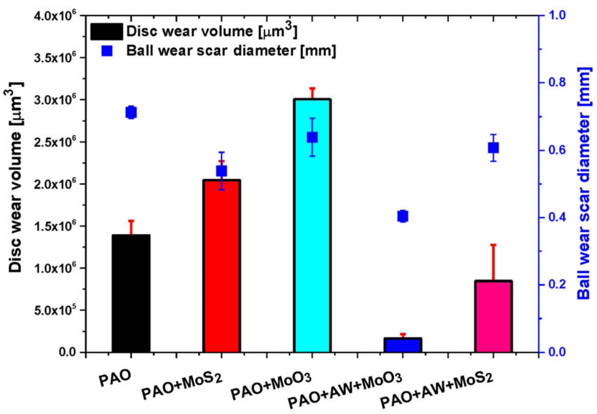


Fig. 3. Disc and ball wear as a function of the lubricant mixture.

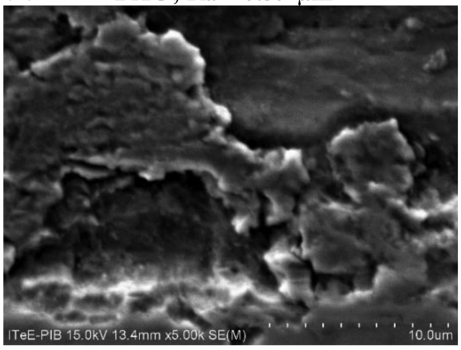
continuously formed and removed, acting as a sacrificial layer. However, this wear protection mechanism comes at a cost of a high friction [15]. Therefore, the presence of a ZDDP in our lubricant blends cannot explain the low friction observed in reciprocating sliding experiments. A particular synergy can be observed between the AW additive and MoO_3 nanotubes, where the lowest wear on the disc as well on the ball can be noticed. For the lubricant blend PAO + AW + MoS_2 also a low wear can be noticed; however it is five times higher than for PAO + AW + MoO_3 due to the sharp friction peaks at the end of the measured temperature, shortly above 200 °C.

3.3. Surface analytics

The formation of the tribofilm for the used blends of lubricant additives was assessed by comparing its surface morphology, surface roughness, and chemical composition by using SEM and EDS (Fig. 4a–e). According to Fig. 4a and EDS chemical analyses of the surface, a rough wear track with many fractures and breaches can be observed. It is typical for steel–steel contacts lubricated with an additive-free PAO base oil. Clearly, the high presence of iron oxides and relatively large amounts of Cr and Mn indicate that there is no continuous tribofilm on the steel surface. This state of the surface is not surprising when we go back to the friction scans presented in Fig. 2a, showing that with the PAO base oil the friction was very high, reaching during the first 2 min of the test a value of 0.45 (lubricant film broke), where the deepest craters were formed, and stabilizing in a later stage of the test to the value of 0.2.

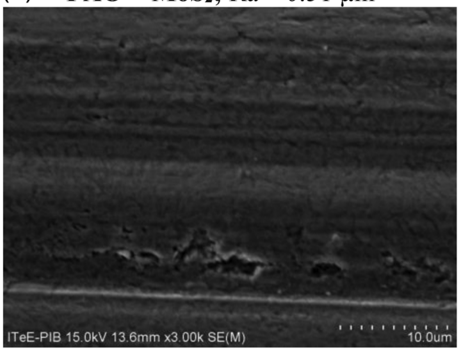
Whenever the PAO base oil was blended with additives (NTs or AW + NTs) not only the friction was much lower (Fig. 2a) but also the quality of the wear tracks surface was much better as shown in Fig. 4b–e. The roughness value within the wear track dropped from 0.59 μm down to 0.51 μm after the test with PAO and to 0.29 μm after the tests with PAO + NTs, and the surface morphology was fine with visible typical patches of the tribofilm formed from NTs (Fig. 4b, c) as shown also by Tomala et al. [6]. The formation of a thin tribolayer derived from MoS_2 NTs on the steel surface (Fig. 4b) was confirmed with EDS analyses showing presence of 1.3% molybdenum and 0.5% sulphur. A much thicker tribolayer derived from MoO_3 NTs on the steel surface is shown in Fig. 4c, confirmed with analyses showing almost 5% molybdenum and 29% oxygen and only 60% iron. After the tests with pure PAO oil the amount of iron was almost 73%, confirming the thicker coverage of the steel surface after tests with PAO + MoO_3 NTs.

(a) **PAO;** Ra = 0.59 μm



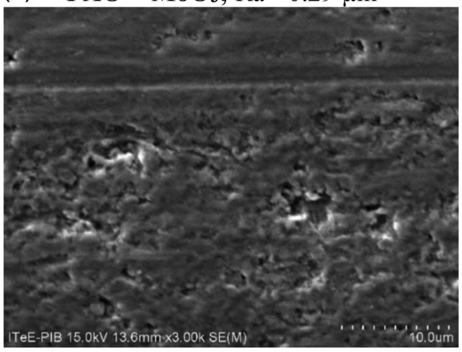
Element	Atom%
C	12.54
O	13.10
Si	0.36
Cr	1.19
Mn	0.14
Fe	72.66

(b) **PAO + MoS₂;** Ra = 0.51 μm



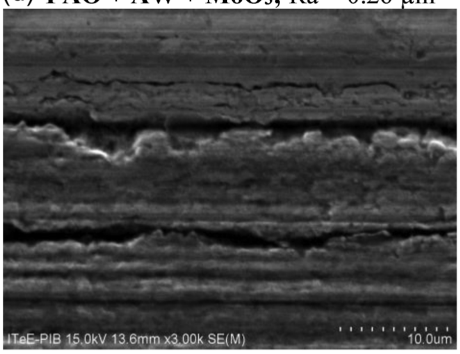
Element	Atom%	Element	Atom%
C	15.30	Cr	0.68
O	9.75	Mn	0.00
Si	0.09	Fe	72.41
S	**0.47**	**Mo**	**1.30**

(c) **PAO + MoO₃;** Ra = 0.29 μm



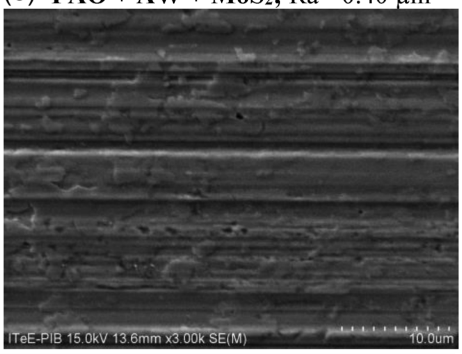
Element	Atom%		
C	5.30	Cr	1.07
O	**28.67**	Mn	0.41
Si	0.39	Fe	59.26
		Mo	**4.89**

(d) **PAO + AW + MoO₃;** Ra = 0.26 μm



Element	Atom%	Element	Atom%
C	7.12	Cr	1.16
O	**22.51**	Mn	0.38
Si	0.16	Fe	45.86
P	**5.72**	**Zn**	**9.51**
S	**6.36**	**Mo**	**1.20**

(e) **PAO + AW + MoS₂;** Ra = 0.40 μm



Element	Atom%	Element	Atom%
C	4.68	Cr	1.55
O	5.60	Mn	0.52
Si	0.38	Fe	83.27
P	**0.54**	**Zn**	**0.90**
S	**1.38**	**Mo**	**1.18**

Fig. 4. SEM/EDS analyses after the SRV reciprocating sliding test of the wear tracks of discs lubricated with (a) PAO, (b) PAO mixed with MoS_2 NTs, (c) PAO mixed with MoO_3 NTs, (d) PAO blended with AW additive and MoO_3 NTs, (e) PAO blended with AW additive and MoS_2 NTs. Boldface highlights the elements responsible for the tribofilm formation. Ra – roughness.

The thickest tribofilm, and at the same time the smoothest surface, was formed after the reciprocating sliding test with the PAO + AW + MoO_3 lubricating blend, which can be inspected in Fig. 4d. The tribotest with this blend exhibited low friction (Fig. 2a, b) and the lowest wear and surface damage (Fig. 3). The morphology of the wear track tested with MoO_3 and AW shows very few signs of wear and subtle abrasive marks seen running parallel to the sliding direction, which indicate the occurrence of mild two-body abrasion. The elements found in the area of this wear track correspond to molybdenum, iron, and oxygen indicating the possible presence of remains of MoO_3 nanotubes over the iron substrate. The high presence of zinc, phosphorus, and sulphur suggests the formation of a ZDDP tribofilm. These observations confirm the excellent performance of this lubricant mixture not only in terms of wear but also friction, and evidence that the superb AW properties of ZDDPs are preserved when used in conjunction with MoO_3 nanotubes. Also Rodrígues Ripoll et al. [11] showed by Raman spectroscopy that in-situ formation of MoS_2 from MoO_3 nanotubes during the sliding contact process in the presence of a ZDDP AW additive is feasible. The in-situ formation of MoS_2 can also be achieved by using MoDTC in combination with a ZDDP as reported by Morina et al. [15].

For comparison, the sample tested using the lubricant mixture of PAO blended with 2 wt% AW additive and 2 wt% MoS_2 NTs is presented in Fig. 4e. This wear track reveals the characteristic presence of patches as a consequence of the tribofilm formed by the exfoliation of MoS_2 lamellae during the sliding contact [4]. The presence of Mo, S, P, and Zn on the tribofilm is evidenced by a weak signal from EDS (Fig. 4e).

4. DISCUSSION

In our experiments the innovative lubricant additives in the form of MoS_2 and MoO_3 nanotubes blended in base oil did not suffer harsh initial running in the process, the tests initiated smoothly and quietly. According to the literature [16], MoS_2 NTs are able to reduce friction by 50–60% and the wear coefficient by 60–90% in room temperature experiments. In our research at elevated and high temperatures (40 to 220 °C), as expected, MoS_2 nanotubes reduced CoF values by 30% compared to the reference oil; nevertheless, the wear at the end of the temperature test was not satisfactory, meaning that MoS_2 NTs underwent oxidization to MoO_3 at higher temperatures, and exfoliation in the tribofilm occurred on the surface. However, MoO_3 NTs alone did not perform satisfactorily, reducing the CoF only slightly, while wear was even higher than in case of the base oil.

Excellent tribological performance was found for the lubricating blends containing both innovative NTs and the traditional ZDDP mixture, indicating strong tribochemical synergy between them. The reason is that under demanding conditions MoS_2 NTs are known to oxidize at the contact interface, while the combination of MoO_3 NTs and sulphur compounds present in ZDDPs leads to a constant sulphurization on demand. The mixture containing MoS_2 NTs and ZDDPs suffered high wear at the end of the tribotest (last 5 min), which means that the temperature of 200 °C caused a dramatic loss of performance.

The findings presented in this article confirm the in-situ sulphurization of MoO_3 NTs mixed with traditional ZDDP AW additives, showing that this additive combination can stand severe oil temperatures and tribotest conditions. The tribochemically formed tribofilm derived from ZDDP and in-situ sulphurized MoO_3 was much thicker compared to other lubricating blends investigated in our research so far. Our approach shows that MoO_3 nanotubes could fulfil the role of the friction modifier in combination with a ZDDP and be able to replace organo-molybdenum compounds, which pose serious environmental concerns. Furthermore, this research can be considered as an alternative approach in lubrication science, which may be beneficial in further industrial applications of oil additives.

5. CONCLUSIONS

- At elevated and high temperatures (40–220 °C) MoS_2 nanotubes reduced the coefficient of friction compared to the reference oil; nevertheless, the wear characteristics were not satisfactory.
- The MoO_3 nanotubes alone did not perform satisfactorily, only slightly reducing the coefficent of friction all over the temperature range studied, while the wear after the test was even higher than for the base oil.
- Only the combination of the traditional ZDDP antiwear additive and the innovative MoS_2 and MoO_3 nanotubes in the base oil provided excellent tribological performance in terms of friction and wear under severe tribotest conditions and the oil temperatures reaching the upper operational limit.
- Particularly strong tribochemical synergy was reached between the MoO_3 nanotubes and the S-rich antiwear additive, indicating the in-situ sulphurization process resulting in the formation of a thick tribolayer due to protective additives.

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Triboloogiline sünergia klassikalise tsink-dialküül-ditiofosfaadi (ZDDP) ja innovatiivsete MoS_2 ning MoO_3 nanotorulisandite vahel kõrgendatud temperatuuridel

Agnieszka Maria Tomala, Manel Rodríguez Ripoll ja Remigiusz Michalczewski

Üheks põhiliseks takistuseks MoS_2 nanotorude õlilisandina kasutamisel on nende temperatuuritundlikkus. Varasemad uuringud näitasid, et MoS_2 nanotorud võivad asendatud olla MoO_3 nanotorudega koos väävlit sisaldavate määrdeainetega, pidades silmas uut lähenemist *in situ* tribokeemilisel väävelrikastamisel. Töö eesmärgiks on uurida temperatuuri mõju innovatiivsete MoS_2 ja MoO_3 nanotorudest määrdelisandite triboomadustele. Nanotorud segati väävlilisandeid sisaldava ja mittesisaldava õliga. Triboomadusi uuriti võnkeliuge katseseadmel teraskuul-terasketas temperatuurivahemikus 40–220 °C. Katsetulemused näitasid traditsiooniliste kulumisvastaste lisandite ja innovatiivsete nanotorude väga positiivset sünergiat, tagades suurepärase triboomaduste paranemise kuni temperatuurini 200 °C. Saadud tulemused näitasid, et kulumise käigus MoO_3 nanotorude väävelrikastamine on edendanud traditsioonilise ZDDP kasutamist kulumisvastase lisandina, määrates selliste lisandite kombinatsiooni stabiilsuse rasketes temperatuuri- ja tribotingimustes. Teiste seni meie uuritud määrdesegudega võrreldes on tribokeemiliselt ZDDP-st moodustunud tribokile ja kulumise käigus väävliga rikastatud MoO_3 tunduvalt paksemad.