NORDTRIB 2018

The 18th Nordic Symposium on Tribology – NORDTRIB 2018
18-21 June 2018, Uppsala University, Uppsala, Sweden

PERFORMANCE OF MoS_2 NANOTUBES BASED LUBRICANT IN STEEL/COATING CONFIGURATION

Agnieszka Tomala[1,2*], Remigiusz Michalczewski [1], Manel Rodriguez Ripoll [2]

[1] ITeE Institute for Sustainable Technologies – National Research Institute, 26-600 Radom, POLAND.
E-mail: agnieszka.tomala@itee.radom.pl, Remigiusz.Michalczewski@itee.radom.pl, Manel.Rodriguez.Ripoll@ac2t.at.
[2] AC2T research GmbH, 2700 Wiener Neustadt, AUSTRIA.

ABSTRACT:

Inorganic Fullerene like nanoparticles, particularly MoS_2 nanotubes (NTs) are developed into innovative lubricant additive to enhance tribological performance of current mechanical components made of steel through superb friction- and anti-wear reduction properties. Nowadays, in modern and safe machines, non-ferrous surfaces are becoming more widely used, mainly as multifunctional coatings working in configuration with steel pair. As a consequence essential in future lubrication technologies and at the same time the purpose of this work is to reveal interaction mechanism of nanoparticles with coated elements in lubricated contacts.

The tribological properties were investigated using reciprocating sliding testing machine in steel ball on coated disc configuration and four-ball testing machine in combination with coated ball on three steel balls. For tribological studies two commercially available coatings were selected: WC/C and DLC coating. Frictional results show only slight improvement for lubricant blends containing MoS_2 nanotubes with selected coatings, surprisingly wear tracks are very extensive when compare to non-additivated oil. Generally, the presence of coating in any tribological test configuration diminish the effectiveness of MoS_2 NTs when compare to steel/steel contact. Chemical analytics show that interaction mechanisms of the nanotubes and surface changes depend on the coating material ability to form chemically derived tribofilm.

Keywords: Nanoparticles, MoS_2 nanotubes, Additives, Tribofilm, Friction, Wear.

INTRODUCTION

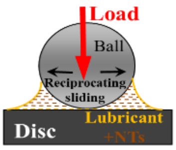
The stringent requirements from environmental legislations on reducing harmful elements is constantly forcing lubricant manufacturers to produce and implement greener additives. In a same way, equipment manufacturers are encourage to develop and implement novel materials in machine elements contacts with better mechanical, wear and frictional properties. Some potential materials that are considered to hold great promise for such objectives and are of paramount interest in this research work are diamond-like carbon (DLC) coatings [1][2]. However, a major challenge involved in using these materials is ensuring satisfactory tribological performance with existing lubricant additives. This is because the existing lubricant additives (*e.g.* AW/EP, friction modifiers, detergents, dispersants, e*tc*) were designed to work with ferrous surfaces (e.g. steel on steel) and no adequate confidence exists on whether these additives can work and effectively reduce friction and wear with non-ferrous surfaces (e.g. DLC coatings) [3]. Our approach to address this issue is to focus on latter aspects, i.e. on novel additives in a form of inorganic fullerene-like (IF) and nano-tubular structures [4]. Using lamellar nanoparticles such as IF-MoS_2 offer a superior lubrication performance due to their low-shear resistance to any applied shear stress [5]. Unfortunately, the tribological performance of these particles is affected by the morphology, structure, size along with the large influence of the test conditions [6]. So far the main conclusion has been that IF-MoS_2 in form of multiwall nanotubes (NTs) contain more defects than the IF-MoS_2 in for of platelets and therefore are easily exfoliated. Due to the shearing of the basal planes in IF particles, it was recently proposed that such a physical based mechanism can be used for the lubrication of materials with lower chemical reactivity e.g. DLC coatings [8]. Attempts at lubricating DLC coatings with oils containing IF nanoparticles have been contradictory so far. From one site it was reported that this combination was not successful [9], while another report showed that under boundary lubrication condition reduction in friction can reach 50% compared to the base oil without MoS_2 nanoparticles [10]. None of the reports address the upper and lower specimens' wear rate, but mainly report on frictional performance.

Accordingly in this work the tribological properties were investigated using reciprocating sliding testing machine in steel ball on coated disc configuration stressing on wear mechanism appearing on two commercially available coatings: WC/C and DLC coating.

MATERIALS AND METHODS

The tribological tests were performed on a SRV® tribometer (Optimol Instruments Prüftechnik GmbH, Germany) under reciprocating sliding conditions using a point of contact at the tests parameters given in Table 1. The base material used tribological tests was AISI 52100 bearing steel. with a microstructure formed by fine martensitic iron and disperse micrometer size carbides, which resulted in a hardness of 850 HV10 and a roughness of Ra 0.05 µm. As counterbody, also AISI 52100 bearing steel balls with a diameter of 10 mm were used with the same hardness and roughness. Another two series of SRV discs were coated at Oerlicon Balzers with BALINIT@ C STAR (CrN (1 µm) + a-C:H:Me (WC/C) (2 µm)) and BALINIT@ DLC (CrN (1 µm) + a-C:H (2 µm)). Both coatings benefit from a hard, durable 1 µm thick metal-based inter layer chromium nitride, which supports the superposed carbon coatings (load-bearing capacity). The thickness of both carbon coatings and the interlayer was measured using Calowear f-my CSM and optical camera VHX1000E Keyence.

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

Tribological test set-up	SRV® test  	
Contact conditions	Point contact at **reciprocating sliding**	
Track length	1 mm	
Tested Disc - Ball materials	Steel - steel DLC - steel	WC/C - steel
Speed	0.1 m/s (50 Hz)	0.04 m/s (20 Hz)
Normal Load	100 N	10 N
Mean contact pressure	1,46 GPa	0,68 GPa
Test duration	60 min	30 min
Temperature	40°C	
Lubricant blends	PAO, PAO+AW, PAO+NTs, PAO+AW+NTs	
Measured parameters	Coefficient of friction vs time, wear rate on discs and balls wear area	

The base oil used in this study was NEXBASE® 2008 polyalphaolefine (PAO) with a viscosity of 24.6 mm²/s at 40 °C and 4 mm²/s at 100 °C. The AW additive used in this study is a mixed primary/secondary zinc dialkyl dithiophosphate ZDDP with 99% purity from Afton chemicals. The MoS_2 nanotubes (NTs) investigated in this study were synthesized from $Mo_8S_2I_8$ nanowires by the procedure reported in [11]. The diameter of the NTs is in the range of 100–150 nm, while their length is up to 3 µm. The walls of the NTs are approx. 10 nm thick and form dome terminations. The lubricant mixtures containing NTs were homogenized using an ultrasonic processor UP200H (Hielscher – Ultrasound Technology). The selected parameters for 10 ml lubricant blend at the probe tip were 20% amplitude during 5 min, while the pulse was on and off for 2 seconds, respectively.

Surface topography of the tested samples was evaluated 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 software was used for wear volume analysis. Optical measurements of the balls wear scare diameter with precision up to 1µm were realized using Nikon MM-40/L3FA.

RESULTS AND DISCUSSION

Friction

In order to understand the effect of lubricants containing MoS_2 nanotubes reciprocating sliding tests were carried out to evaluate the tribological performance of multifunctional coatings working in configuration against steel pair. The SRV results are presented in a form of frictional scan as a function of time (Fig. 1). It is important to note that the error bars presented on the frictional curves represent the average of three repetitions, and it can be stated that reproducibility of the results was very good for all test conditions. As expected and reported previously by many researches [12], strong friction reduction (by 45% compare to the base oil) is observed for the lubricant blend containing MoS_2 NTs in steel - steel material configuration (Fig. 1a). In DLC – steel material combination the effect of the lubricant containing MoS_2 NTs is much lower reaching 24% (Fig. 1b). For the following material configuration WC/C – steel the friction reduction between PAO and PAO+NTs is only 10% (Fig. 1c).

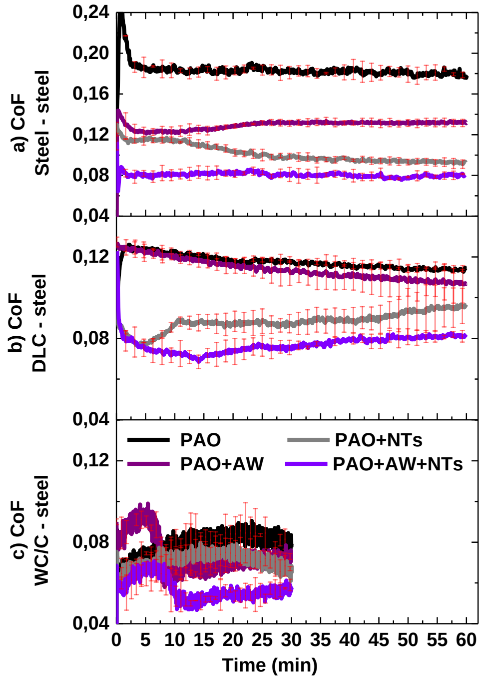


Figure 1. SRV tests results represented with friction curves including error bars distribution over the curve for following material configurations a) steel – steel, b) DLC – steel, c) WC/C – steel.

Addition of AW additive to the PAO influenced the base oil performance in terms of friction the strongest in steel-steel test configuration (Fig.1a). In DLC – steel contact there is no effect (Fig.1b), and in WC/C – steel configuration transition from higher friction in first 10 min of the test to the lower friction after that time has been observed (Fig.1c). The best results in term of friction for all materials configuration was observed for PAO+AW+NTs lubricating blend. Friction reduction was 56% in steel-steel contact, 34% in DLC-steel and 27% in WC/C-steel disc-ball material arrangement. The synergistic effect between MoS_2 NTs and ZDDP additive could also be highlighted within the present work and is in agreement with results reported in literature [13].

The reason is formation of nanosheets within the tribofilm formed on bare steel surface (iron oxide), and within/on the top layer of ZDDP tribofilm formed on steel surface with preformed ZDDP film. The mechanism on coated surfaces is not yet revealed.

Wear

Specific wear rates on the disc were calculated according the Archard's formula: $K = \frac{V}{F \times S}$ where V is the wear volume (μm^3), F is the normal load (N), and S is the sliding distance (m). Balls wear area were calculated from the formula $\frac{\pi \times d1 \times d2}{4}$ where d_1 is horizontal wear diameter and d_2 is vertical wear diameter.

As visible in Fig. 2 in reciprocating sliding point contact the most sensitive for wearing is WC/C coating. Due to this fact the parameters of the test were reduced to the minimum, where the interactions of the lubricant blends and the WC/C coating were strongest, without reaching the CrN interlayer.

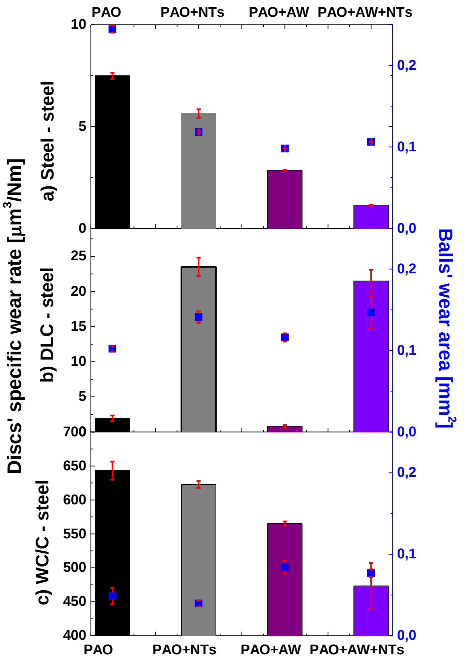


Figure2. Disc and ball wear of the different tested pairs as function of the lubricant mixture.

As observed in Fig. 2a on the steel-steel surfaces, the disc and the ball wear with additive-free PAO was the highest, which however was reduced when NTs were used. The strongest reduction of wear rate was obtained for PAO+AW and PAO+AW+NTs, which is not surprising since ZDDP forms thick tribofilms that prevent surfaces from wearing. It is interesting to note that similarly like in frictional results the best synergy on the steel surface is observed for PAO+AW+NTs lubricant blend. In this case the original anti-wear properties of the formed ZDDP tribofilm are further improved, what suggest that NTs were incorporated within and deposited on the top of ZDDP layer building even thicker additives derived tribofilm. Similar effect of the additives was observed on the WC/C – steel tested materials (Fig. 2c).

However on DLC – steel material configuration visible in Fig.2 b reverse action of the lubricant blend containing NTs can be observed. Only when MoS_2 NTs are used either accompanied with AW or not extensive wear is visible on DLC surface, suggesting that NTs are scrubbing this coating. The AW additive represented by ZDDP similarly like on steel surface forms thick tribofilms that prevent surfaces from wearing. Nonetheless adding NTs to the PAO+AW blend diminish the effectiveness of ZDDP.

CONCLUSIONS

- In terms of friction the best results were observed for PAO+AW+NTs lubricating blend for all materials configuration, suggesting strong synergetic effect between ZDDP additive and MoS_2 nanotubes.
- Steel surface and DLC coating are most sensitive for action of additives. WC/C seems to less sensitive to the lubricant mixture.
- In terms of wear, the best protection was spotted for PAO+AW+NTs blend except for DLC coatings.
- DLC coatings exhibit high wear for both lubricant blends containing MoS_2 NTs.

REFERENCES

[1] K. Holmberg, H. Ronkainen, A. Laukkanen, and K. Wallin, "Friction and wear of coated surfaces — scales, modelling and simulation of tribomechanisms," *Surf. Coatings Technol.*, vol. 202, no. 4–7, pp. 1034–1049, 2007.

[2] R. Michalczewski, W. Piekoszewski, M. Szczerek, W. Tuszynski, and M. Antonov, "The Rolling Contact Fatigue of PVD Coated Spur Gears," in *Engineering Materials and Tribology*, 2013, vol. 527, pp. 77–82.

[3] M. Kalin, I. Velkavrh, J. Vižintin, and L. Ožbolt, "Review of boundary lubrication mechanisms of DLC coatings used in mechanical applications," *Meccanica*, vol. 43, no. 6, pp. 623–637, 2008.

[4] R. Tenne, L. Margulis, M. Genut, and G. Hodes, "Polyhedral and cylindrical structures of tungsten disulphide," *Nature*, vol. 360, no. 6403, pp. 444–446, Dec. 1992.

[5] J. M. Martin, C. Donnet, T. Le Mogne, and T. Epicier, "Superlubricity of molybdenum disulphide," *Phys. Rev. B*, vol. 48, no. 14, pp. 10583–10586, Oct. 1993.

[6] I. Lahouij, B. Vacher, J. M. Martin, and F. Dassenoy, "IF-MoS2 based lubricants: Influence of size, shape and crystal structure," *Wear*, vol. 296, pp. 558–567, 2012.

[7] L. Joly-Pottuz and F. Dassenoy, "Nanoparticles Made of Metal Dichalcogenides," in *Nanolubricants*, 2008, pp. 15–92.

[8] J. Kogovšek, M. Remškar, and M. Kalin, "Lubrication of DLC-coated surfaces with MoS2 nanotubes in all lubrication regimes: Surface roughness and running-in effects," *Wear*, vol. 303, no. 1–2, pp. 361–370, 2013.

[9] J. Tannous, F. Dassenoy, I. Lahouij, T. Le Mogne, B. Vacher, A. Bruhács, and W. Tremel, "Understanding the tribochemical mechanisms of IF-MoS 2 nanoparticles under boundary lubrication," *Tribol. Lett.*, vol. 41, no. 1, pp. 55–64, 2011.

[10] M. Kalin, J. Kogovšek, J. Kovač, and M. Remškar, "The Formation of Tribofilms of MoS2 Nanotubes on Steel and DLC-Coated Surfaces," *Tribol. Lett.*, vol. 55, no. 3, pp. 381–391, 2014.

[11] M. Remškar, M. Viršek, and A. Mrzel, "The MoS2 nanotube hybrids," *Appl. Phys. Lett.*, vol. 95, no. 13, pp. 2–4, 2009.

[12] A. Tomala, M. R. Ripoll, C. Gabler, M. Remškar, and M.

Kalin, "Interactions between MoS2 nanotubes and conventional additives in model oils," *Tribol. Int.*, vol. 110, 2017.

[13] A. Tomala, B. Vengudusamy, M. Rodríguez Ripoll, A. Naveira Suarez, M. Remškar, and R. Rosentsveig, "Interaction Between Selected MoS<inf>2</inf> Nanoparticles and ZDDP Tribofilms," *Tribol. Lett.*, vol. 59, no. 1, 2015.

ACKNOWLEDGEMENTS

This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 665778. The author A. Tomala acknowledges the POLONEZ project by National Science Centre, Poland under fellowship registration number 2015/19/P/ST8/02597. This work was funded by the Austrian COMET Programme (Project K2 XTribology. No. 849109).