

Effect of Temperature on the Tribological Performance of MoS₂–TiO₂ Coating Material



Avinash V. Borgaonkar and Ismail Syed

Abstract Recently, there is a growing interest in molybdenum disulphide (MoS₂) to use as a solid lubricant to enhance the tribological properties. In the present work, composite MoS₂/TiO₂ coatings were prepared and bonded on pre-treated steel substrate. The manganese phosphating process has been used as a pre-treatment which helps to improve the bond strength. The coating has been bonded onto the phosphated substrate using sodium silicate as a binder. A comparative study of these composite coatings was done at different temperature, load and speed conditions using pin-on-disc tribometer. The results depict that the tribological performance such as friction coefficient and specific wear rate has been improved by coating material as compared to uncoated material.

Keywords Molybdenum disulphide · Solid lubricant · Coating · Tribological properties

1 Introduction

Solid lubricants have been proven to be such materials that despite of its solid phase, they are able to provide improved tribological performance between the rolling/sliding interfaces. The solid lubricant can be either used as coatings or as filler element in self-lubricating composites [1]. From the past few decades, they have been used as a lubricant between the contact surfaces subjected to rolling and sliding movement. There are certain applications where only solid lubricants can be used, i.e. in severe conditions and in hostile environments such as high load and high temperature, ultra-low vacuum and cryogenic temperatures, strong radiation, dry and clean lubrication, resistance to contamination and protection against corrosion. The solid lubricants with their classification are as shown in Table 1 [2, 3].

From the past few decades, most of the researchers concentrated on MoS₂ as a solid lubricant due to its weak interatomic forces between the sulphide anions and strong

A. V. Borgaonkar · I. Syed (✉)
Department of Mechanical Engineering, NIT Warangal, Warangal, Telangana 506004, India
e-mail: syedismail7@nitw.ac.in

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Table 1 Classification of solid lubricants

Classification	Key examples
Lamellar solids	Molybdenum disulphide
	Tungsten disulphide
	Hexagonal boron nitride
	Graphite
	Graphite fluoride
	Boric acid
	Monochalcogenides (GaSe, GaS, SnSe)
Soft metals	Silver
	Lead
	Gold
	Indium
	Tin
Mixed oxides	CuO–Re ₂ O ₇
	CuO–MoO ₃
	PbO–B ₂ O ₃
	CoO–MoO ₃
	Cs ₂ O–MoO ₃
	NiO–MoO ₃
	Cs ₂ O–SiO ₂
Single oxides	Re ₂ O ₇
	MoO ₃
	TiO ₂
	ZnO
Halides and sulphates or alkaline earth metals	CaF ₂ , BaF ₂ , SrF ₂
	CaSO ₄ , BaSO ₄ , SrSO ₄
Carbon-based solids	Diamond
	DLC
	Glassy carbon
	Hollow carbon nanotubes
	Fullerenes
	Carbon–carbon and carbon–graphite-based composites
Polymers/organic materials	Zinc stearate
	Waxes
	Soaps
	Polytetrafluoroethylene

covalent bonds within molybdenum. Moreover, it is stable and unaffected by oxygen and diluted acids. As like graphite, it is also having asperity healing attributes, which helps to lower the friction values. On the other hand, it does not require any water vapour or moisture to provide the results which makes it suitable for vacuum applications. Shankara et al. [4] investigated the tribological performance of bonded pure MoS₂ as well as composite MoS₂-zirconia and MoS₂-graphite coating on steel substrate using sodium silicate as a binder. The test results reveal that in comparison with pure MoS₂, composite MoS₂-zirconia improves the tribological performance since zirconia is a ceramic material having high wear resistance. Beside MoS₂-graphite also helps to enhance the tribological performance since by nature graphite is a very soft material [5]. Another study conducted by Ye et al. [6] used novolac epoxy resin as a binder to bond MoS₂ with the substrate. They observed significant improvement in tribological behaviour at relatively higher load range, since the sand blasting which has been used as a pre-treatment helps to improve the adhesion strength. Further with this same coating and binder material, Luo et al. [7] conducted the rotational fretting wear test. The findings were similar to the previous study. However, they observed that the wear mechanism was the combination of delamination, abrasive wear and tribo-oxidation. Tang et al. [8] examined the tribological performance of aramid fibre filled polyamide six different composites with and without addition of nano-MoS₂ under dry sliding contact conditions. They observed that the tribological properties enhanced significantly due to addition of nano-MoS₂. Two main wear mechanisms such as micro-ploughing and plastic deformations were identified. The decrease in wear rate of a coating was observed with increase of aramid fibre content.

Hamilton et al. [9] conducted number of experiments on different five thin-film MoS₂ coatings such as MoS₂-Ni, MoS₂-Ti, MoS₂-Sb₂O₃, MoS₂-C-Sb₂O₃ and MoS₂-Au-Sb₂O₃. In order to study the macroscopic effect of temperature on the tribological properties, the temperature was varied from -80 to 180 °C. The decrease in COF values has been observed with increase in temperature since the thermally activated coatings exhibit stable interfacing layers due to which ultra-low wear occurs, whereas athermal behaviour leads to high rate of wear. Nowadays, the solid lubricants used at high-temperature conditions since most oils, greases and few organic polymers were oxidatively unstable for some appreciable time. Some of the materials can be used as self-lubricating composites and coatings such as dichalcogenides, graphite and graphite fluoride, polyimides, soft oxides, oxidatively stable fluorides and hard coatings to enhance the tribological performance of the contacting surfaces [10]. Zhang et al. [11] added Ag nano-particles to the MoS₂ in order to improve the tribological properties as well as the load bearing capacity since the addition of Ag nano-particles enhances strength of the tribofilm. Moreover at higher temperature, the composite coating demonstrates prolonged life as compared to conventional MoS₂ coating because of the formation of silver molybdate layers. Theiler et al. [12] examined the tribological performance of MoS₂/ta-C bilayer coatings under different environments such as vacuum and air. In order to improve the bonding, a thin chromium interlayer has been introduced. The tribological performance of the coating found to be significantly improved in humid air environment since the coating deposited by laser-arc has relatively high roughness which enhances the endurance

of MoS₂. Recently, Torres et al. [13] in 2018 from their extensive study summarized that from the transition metal dichalcogenides group MoS₂ being a cost-effective and the outstanding tribological performance in dry air and vacuum environment, since it possesses the weak van der Waals adhesion forces between sulphur-like atoms, leading to the formation of easy-to-shear lamellas. Further, Meng et al. [14] studied the tribological performance MoS₂ coating under dry sliding motion at high temperatures with different loads and speeds. It has been observed that MoO₃ results as an oxidation of MoS₂ (i.e. when working temperature is higher than the transition temperature) leads to lower the tribological performance. Renevier et al. [15] deposited MoS₂/Ti (MoST) coating on machine tools to study the tribological behaviour. The results reveal that the tribological performance of the conventional MoS₂ coating has been significantly improved with the addition Ti since Ti possesses high resistance to wear.

From the literature, it has been observed that MoS₂ as a single as well as in combination with other different materials has been used as coating material at ambient and high-temperature applications. However in the previous literature, nobody addressed the use of composite MoS₂/TiO₂ as a solid lubricant. Hence in this paper, the attempt has been made to analyse the performance composite MoS₂/TiO₂ coating material at ambient and high-temperature conditions.

The objective of the present work involves the evaluation of the tribological performance of composite MoS₂/TiO₂ coating on the steel substrate at ambient and high-temperature conditions. The study has been carried out at different temperatures including room temperature (RT), 100, 200, 300 and 400 °C. At the end, the tribological properties have been evaluated for summarizing the study.

2 Methodology

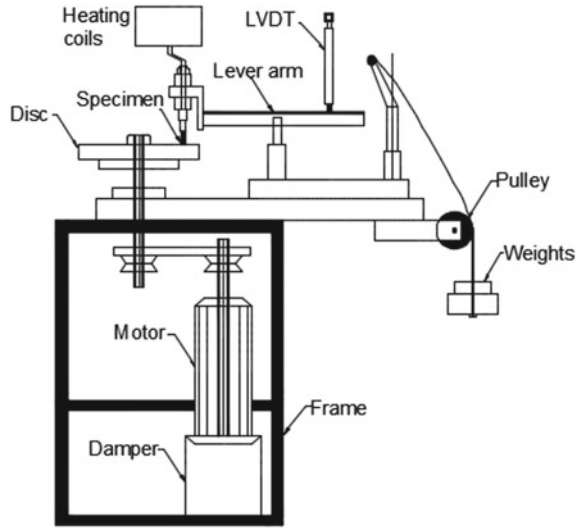
The pin samples were made of AISI 52100 steel (12 mm diameter and 25 mm length) used as the substrate. The AISI 52100 steel composed of 1% C, 1.5% Cr, 0.3% Mn, 0.25% Si, 1% C, with Fe as remaining. Before the experimental test, specimens were polished with SiC abrasive paper (from 220 to 1000-grit), cleaned ultrasonically using acetone and then dried. In order to improve bond strength, manganese phosphating employed as a pre-treatment for the steel substrates. The composite MoS₂/TiO₂ coating applied on the phosphated substrates with the brushing and cured at 150 °C for two hours.

The friction tests were performed using pin-on-disc tribometer (Magnum, India) in accordance with ASTM standard G99-95 [16]. A schematic set-up of the pin-on-disc tribometer is shown in Fig. 1.

The counter disc is made up of EN31 steel. Before performing each test, the disc surface was cleaned using acetone and thoroughly dried. The sliding speed was kept constant by adjusting the rotational frequency and track radius using the formula:

$$V = 2\pi r * N \quad (1)$$

Fig. 1 Experimental set-up for tribological study



where V is the sliding velocity, N is speed in rpm and r is the track radius. A data acquisition system was coupled to the tribometer to compute and record automatically, the tribological parameters between the coated specimen and the disc surface all the time of the experiments. The wear rate can be obtained using the formula:

$$W_R = W_V / P * D \tag{2}$$

where W_R is the specific wear rate in mm^3/Nm , is the wear volume in mm^3 , P is the applied load in N and D is the sliding distance in m. The wear volume was estimated by taking the weights of the samples before and at the end of wear test. Eventually to get the reliable data, each experiment was repeated three times.

The detailed test parameters and ranges are summarized in Table 2.

Table 2 Test parameters and ranges

Sr. No.	Parameters	Operating conditions
1	Normal load	176, 442 and 707 kPa
2	Sliding speed	2, 4 and 6 m/s
3	Track radius	30 mm
4	Sliding distance	3000 m
5	Temperature	RT, 100, 200, 300 and 400 °C
6	Relative humidity	70 ± 5%

3 Results and Discussion

The pin-and-disc surfaces have been analysed before and after wear test to understand the tribological behaviour of the composite coating. The results show that at low load, the mechanism of wear is abrasion, and as the load increases, the coating material from the pin surface gets transferred to the disc surface. So with the increase in load and speed, the mechanism of wear changes from abrasion to adhesion as depicted in Fig. 2.

The experimental test performed at three different load and speed combinations varying the temperature from RT to 400 °C with a step of 100 °C. The values of coefficient of friction (COF) and specific wear rate (mm^3/Nm) for different combinations have been obtained from the data acquisition system and plotted as shown in Figs. 3 and 4.

From the experimental results, it has been observed that friction coefficient (COF) decreases with increase in the temperature and load. At high load with increase in temperature, the coating layer worn out and transferred to the counter disc surface, due to which the reduced values of COF have been observed. The lowest COF value obtained is 0.065. Beside with increase in the temperature and load, the specific wear rate increases, and in the temperature range 300–400 °C, the values of specific wear rate found to be almost consistent. The lowest value of specific wear rate obtained is $1.7 \text{ mm}^3/\text{Nm}$.

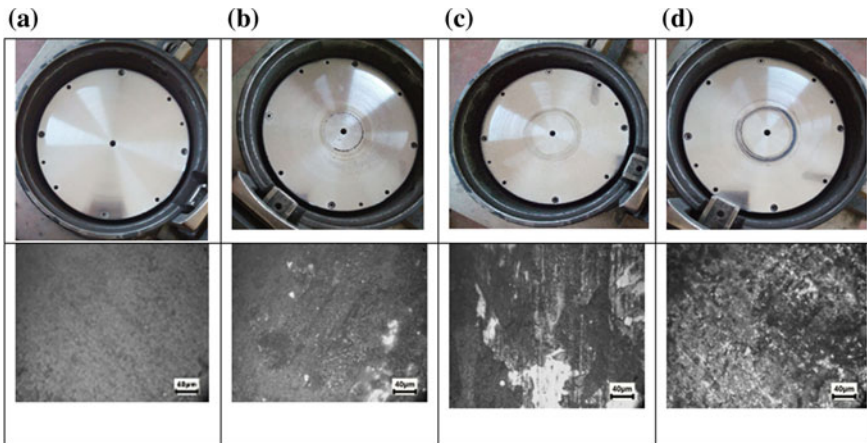


Fig. 2 Disc and pin surface before test (a) and after test at constant load 176 kPa and different sliding speed 2 m/s (b), 4 m/s (c), 6 m/s (d)

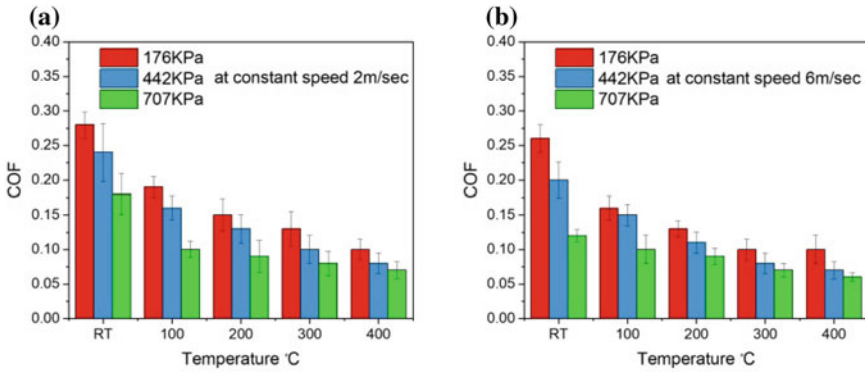


Fig. 3 Coefficient of friction (COF). **a** At load 176, 442 and 707 kPa at constant low speed 2 m/s, **b** at constant high speed 6 m/s

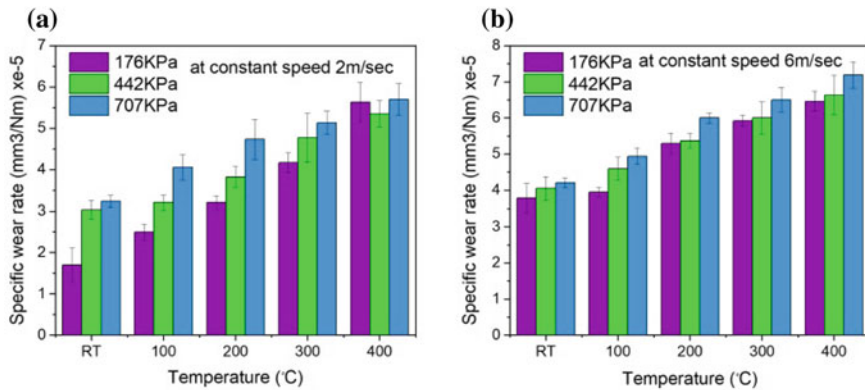


Fig. 4 Specific wear rate (mm³/Nm). **a** At load 176, 442 and 707 kPa at constant low speed 2 m/s, **b** at constant high speed 6 m/s

4 Conclusion

Within the scope of this study, the tribological behaviour of composite of MoS₂/TiO₂ coating at ambient as well as high temperature has been investigated. The results show that due to the synergistic effect of MoS₂ and TiO₂ at high temperature and high load, the friction coefficient was lower. However, due to the change in mechanism from abrasion to adhesion, the coating layers transformed from substrate surface to the counter disc surface with rise in temperature and load, which leads to increase in the specific wear rate. At higher temperatures (i.e. 300–400 °C), the specific wear rate found to be almost constant.

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