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Research article

The Technological Factors and Operating Conditions Influence on the Molybdenum Disulfide Coatings Tribological Properties

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A B S T R A C T

Molybdenum disulfide is a widely used solid lubricant operates successfully under extreme conditions such as vacuum, high or low temperatures, high loads etc. Magnetron sputtering is one of the most common method of application MoS2 coatings. It allows one to apply thin antifrictional films with high rate of purity on different substrates. The surface before application is usually prepared by abrasive processing. The aim of the work is to evaluate the magnetron deposition modes, substrate surface preparation, substrate roughness on the molybdenum disulfide coatings tribological properties. For the purpose mentioned a linearly reciprocating Ball-on-Flat (ASTM G133) Sliding Wear tests in vacuum (pressure less than 10-4 mbar) and high temperature (250°C) were conducted. The two different materials as a coating substrate was used (stainless steel and brass). The substrate surface was sandblasted. The coating surfaces structure after wear and chemical composition have been studied. The friction coefficient in vacuum at 250 °C was about 0.02–0.05. The least value was observed for the AISI 316L steel substrate treated by sandpaper and Ra 0.10 μm roughness coatings. The negative bias voltage (–20 V) during magnetron deposition leads to 16–42% lower coefficient of friction. Oxygen decreases the anti-friction properties.

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1. INTRODUCTION

Molybdenum disulfide (MoS_2) is a solid lubricant that is widely used in the operation of various friction units under extreme conditions. Such conditions include high or low temperature, vacuum, high contact loads, radiation, corrosion. The listed factors that complicate the work of friction units are found in various fields: in space, thermonuclear reactors, cryogenic technology, metal processing, etc. [1].

The lubricating effect of molybdenum disulfide based on its structure. The layers of molybdenum and sulfur atoms are disposed one by one. The atoms are firmly connected by means of a covalent

bond inside the layers, and the forces of intermolecular interaction (van der Waals forces) act between the layers. The latter provide easy relative sliding of the layers and reduce friction [2].

The molybdenum disulfide tribological properties are significantly affected by the content of atomic oxygen, humidity and temperature. MoS_2 coatings can withstand a temperature increase up to 500 ^{0}C in a vacuum or in an inert gas environment. While temperature increases molybdenum oxidation is observed in an oxygen-containing environment. That leads to an increase in the friction coefficient and destruction of the coating. Water molecules disrupt the sliding of the solid lubricant layers in a humid environment, and the coefficient of friction increases. Water molecules, getting into the coating based on molybdenum disulfide during its application, storage or transportation, degrade the anti-friction properties even in a vacuum or an inert gas environment where no moisture is observed [1].

Magnetron sputtering is widely used to apply antifriction solid lubricant coatings based on molybdenum disulfide. Although magnetron deposition requires special vacuum equipment, it allows obtaining thin films of high-purity lubricant uniformly distributed over the coating surface [3].

Sputtering modes and surface preparation have a significant impact on the antifriction MoS_2 coatings tribological properties [4].

The purpose of this work is to determine the magnetron sputtering modes and surface preparation on the molybdenum disulfide coatings tribological properties.

Tribological tests of coatings on bronze CuAl9NiFe4Mn1 and steel AISI 316L substrates were carried out, the structure and chemical composition were studied.

2. MATERIALS AND METHODS

The samples studied are metal plates 40x20x4 mm coated with molybdenum disulfide. The samples characteristics are presented in table 1.

The plates surface was treated with P800 grinding paper, sandblasted with an abrasive material size of 100–150 μm, or left untreated in its original state after rolling, before applying the antifriction coating.

The prepared samples were purged with nitrogen, cleaned in an ultrasonic bath with alcohol, and dried in air, and then were cleaned by a glow discharge in an inert gas atmosphere (glow discharge voltage – 1500 V, argon pressure $2 \cdot 10^{-2}$ mbar).

The molybdenum disulfide coating was obtained using a 3GABS magnetron (Pinch LLC) from a pure MoS_2 target (99.95% purity) in vacuum (10^{-2} mbar).

A constant bias voltage (+100 or –20 V) was applied to the samples during deposition. After the deposition, the samples were cooled for 15 minutes in an argon flow.

The thickness of the applied coating and the roughness of the samples were determined using a Veeco Dektak 150 profiler.

Tribological tests were performed in accordance with ASTM G133 in reciprocating motion. The test bench is equipped with a vacuum chamber and a sample heating system [5]. The coefficient of friction was determined in vacuum ($<10^{-4}$ mbar), at a temperature 250 °C in the friction zone, the sample displacement amplitude was 5 mm, the frequency 30 min^{-1}, test duration 30 min, normal load 7 N. The ball specimen was a diameter of 6.35 mm made of steel 100Cr6. Each coating was tested 3 times.

The coatings and friction surfaces structure and chemical composition were studied using a Tescan Vega 3 scanning electron microscope.

Table 1. Samples characteristics.

Sample	Base material	Surface treatment	Bias voltage, V	Thickness, μm	Ra, μm
A1	AISI 316L	Grinding paper P800	+100	6.0	0.10
A2			-20	6.6	0.10
B1		No	+100	5.6	0.07
B2			-20	7.2	0.07
C1		Sandblasting	+100	6.8	1.46
C2			-20	6.7	2.32
D1	CuAl9NiFe4Mn1	Grinding paper P800	+100	6.6	0.16
D2			-20	5.6	0.12

3. RESULTS AND DISCUSSION

Coatings microstructure (Figure 1) and coefficient of friction diagrams during the tribological tests (Figures 2-5) were obtained.

The coatings surface texture repeats the substrate texture. Traces of abrasive treatment are observed on the A1 sample surface after deposition.

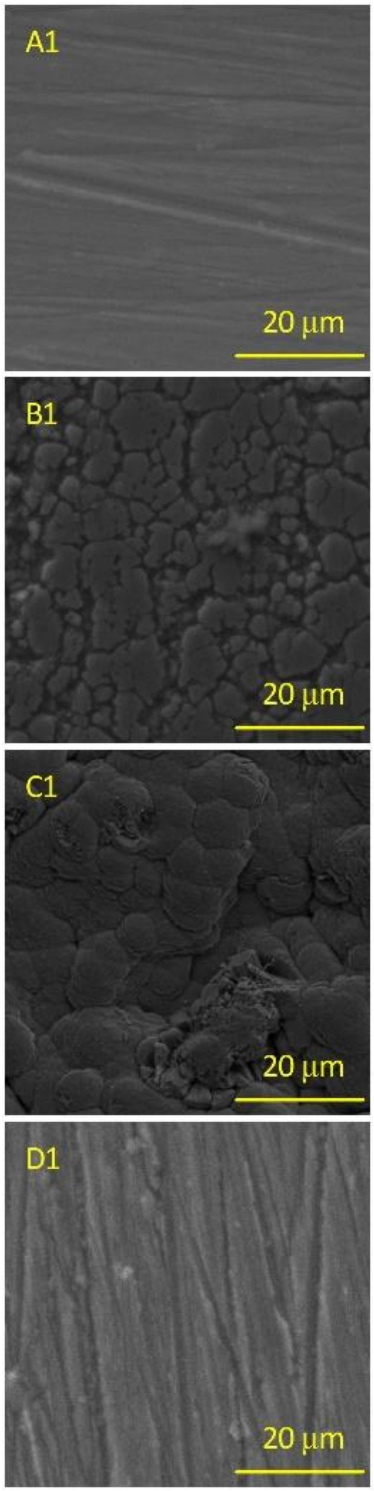


Fig. 1. Samples microstructure.

The initial substrate (after rolling) applied coating (B1) has a granular structure with noticeable discontinuities. The surface sandblasted sample coating (C1) has a globular structure with sputtering direction layered particles. The coating deposited on a bronze substrate (D1) sample structure processed with grinding paper contains traces of abrasive processing on the surface, as well as sample A1.

The friction coefficient of steel specimens treated with abrasive paper is shown in Figure 2. It can be seen that at the beginning of the test, for about 10-12 min, the specimens are running in until the minimum friction coefficient is reached, and then the test is in steady-state mode.

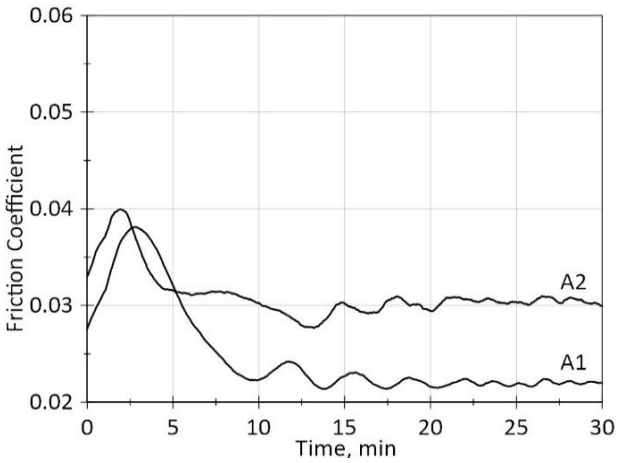


Fig. 2. A1 and A2 sample tribological tests.

The steady state friction occurs after 4-5 minutes with a coefficient of friction slightly different from the value at the beginning in tribological tests of coatings B1 and B2 (Figure 3).

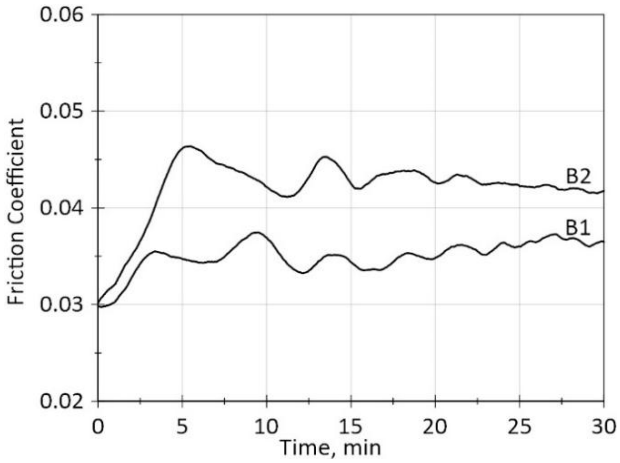


Fig. 3. B1 and B2 sample tribological tests.

The steady-state friction mode was not reached during the test of sandblasted specimens C1 and C2 (Figure 4). A gradual increase in the friction coefficient was observed while the process of running-in.

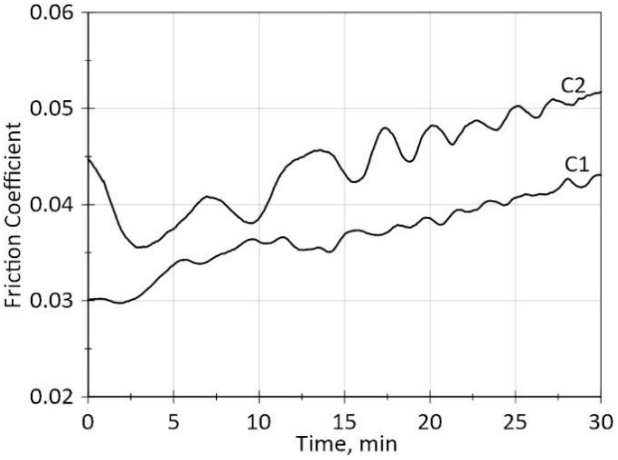


Fig. 4. C1 and C2 sample tribological tests.

The D1 and D2 samples friction coefficient (Figure 5) slightly increased from the initial value during running-in and then switched to the steady-state friction mode after about 14-15 min.

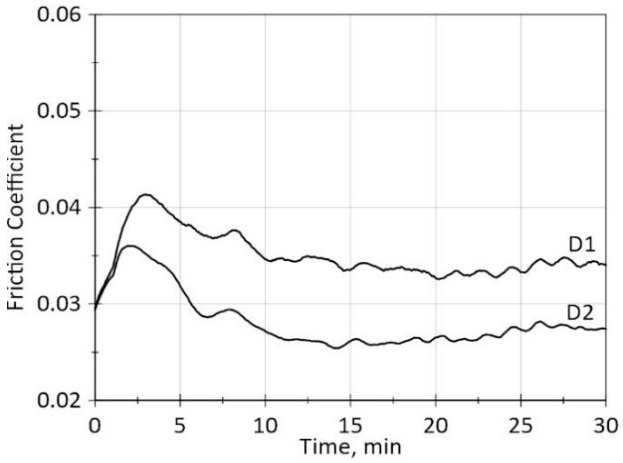


Fig. 5. D1 and D2 sample tribological tests.

The coatings main chemical elements ratio before and after tribological tests the coefficient of friction (CoF) are shown in Table 2.

The relative oxygen content in all samples increased after tribological tests. A higher oxygen content in the coating before testing for all samples except D1 and D2 is accompanied by a higher value of the coefficient of friction.

The vacuum deposited molybdenum disulfide coatings friction coefficient at a 250°C temperature was 0.02–0.05.

The friction coefficient of coatings deposited on a steel substrate (A, B and C) with a negative bias voltage (-20 V) is 16-42% lower than that obtained at a voltage of +100 V.

Table 2. Chemical elements ratio.

Sample	Before tests		After tests		CoF
	O/S	S/Mo	O/S	S/Mo	
A1	0.10	1.74	0.15	1.59	0.022
A2	0.13	1.71	0.18	1.64	0.030
B1	0.12	1.69	0.17	1.62	0.036
B2	0.21	2.02	0.27	1.8	0.042
C1	0.1	2.09	0.22	1.91	0.042
C2	0.11	2.01	0.16	1.93	0.051
D1	0.10	1.69	0.23	1.61	0.035
D2	0.11	1.71	0.18	1.66	0.028

The surface preparing method for the spraying samples with a steel substrate affects the value of the friction coefficient. Processing with sandpaper increases the surface roughness to Ra 0.10 μm compared to untreated. This leads to a decrease in the coefficient of friction. Further increase in roughness by sandblasting, on the contrary, rises the coefficient of friction. Gradt at al. [6] show that the minimum friction is achieved at a roughness value in the range of Ra=0.10–0.15 μm.

Since the tribological tests were carried out in a vacuum, an increase in the oxygen content in the coating after tribological tests can occur as a result of the oxygen accumulation during storage and transportation of the samples. Or while the sample were cooled in air after tests.

The tribological tests results for samples deposited on a bronze substrate are different. Obtained at a negative bias voltage sample (D2) contains a higher oxygen content and has a friction coefficient 25% lower compared to D1. However, the roughness of sample D1 (Ra 0.16 μm) is higher than that of D2 (Ra 0.12 μm), which could affect the friction coefficient.

4. CONCLUSION

The magnetron sputtered molybdenum disulfide coatings have good antifriction properties (friction coefficient 0.02–0.05) in vacuum (pressure less than 10^{-4} mbar) at a temperature of 250 °C. The coatings with an AISI 316L steel substrate treated with sandpaper to a Ra 0.10 μm roughness showed least friction coefficient under these test conditions. the friction coefficient of the friction coefficient of the samples with a negative bias voltage (–20 V), applied to the substrate during magnetron deposition, under these test conditions is 16–42% lower. Oxygen contains in the coating degrades the anti-friction properties.

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