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The Influence of Substrate, Binder, and Additives on Suspension Coating Properties at Elevated Temperatures

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Abstract: A study of the antifriction properties of suspension solid-lubricating coatings based on molybdenum disulfide (MoS_2) at high temperatures depending on the type of substrate, binder, additives, and load parameters was carried out. The solid lubricants were sprayed on two different substrates, high-temperature alloy (Inconel X-750) and stainless steel (AISI 430), tested under 10 N and 23 N loads at temperatures ranging from 25 °C to 800 °C. For comparison, different types of solid lubricants were used. In this work, it was established that the antifriction properties of solid lubricant suspension coatings at high temperatures significantly depend on the type of solid lubricant and the binder used. Moreover, it was shown that the use of Inconel X-750 as a substrate can lead to an increase in the critical operating temperature of coatings containing MoS_2, graphite, and titanate as solid lubricant, additive, and binder, respectively. For instance, at load 23 N, the operating temperature increased from 480 °C to 496 °C. On the other hand, the coating based on graphite, containing ceramic as an additive, and an inorganic binder showed the best performance in terms of a combination of properties (low coefficient of friction and longer operation with a coefficient of friction below 0.3 under increasing temperature) when it was applied on the Inconel X-750 substrate. In addition, it was established that the coefficient of friction of graphite-based coatings gradually increases as they lose their antifriction properties due to their failure, while the coatings based on molybdenum disulfide show the opposite behavior, where the coefficient of friction increases sharply when it loses its lubricating properties.

Keywords: coatings; antifriction properties; tribology; MoS_2; wear; solid lubricant

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1. Introduction

Currently, various types of coatings are used to enhance the tribological properties of friction units obtained by methods such as chemical deposition from the gas phase (CVD) or physical deposition from the gas phase (PVD), as well as coatings based on solid lubricants, which often consist of graphite or molybdenum disulfide (MoS_2) [1–3]. The lubricating effect of molybdenum disulfide is due to its layered structure, which is similar to that of graphite. In this structure, molybdenum and sulfur atoms form strong covalent bonds, and the MoS_2 layers are connected by weak van der Waals forces, resulting in low shear resistance relative to each other [4,5].

However, layered materials such as molybdenum disulfide usually oxidize in air at about 300 °C, which limits their use at high temperatures. In cases where a lubricant operating at temperatures above 300 °C is required, tungsten disulfide (WS_2) is used. This is due to the fact that its upper operating temperature limit is about 100 °C higher than that of MoS_2 [6].

In a vacuum or in an inert gas environment, MoS_2 exhibits a low coefficient of friction (μ) (less than 0.05), high performance, and reliable lubrication lasting for several million cycles, even at high temperatures of up to 400–500 °C [4,7,8]. As the temperature increases, water desorption occurs from the surface of the antifriction layer and friction decreases. This effect of reducing friction with increasing temperature is observed up to 100 °C. The process of increasing friction during the adsorption of water molecules is thermally reversible [7,9,10]. The requirements for friction units are constantly increasing. When the temperature rises to 100–400 °C in the presence of oxygen, the surface of molybdenum disulfide films starts to oxidize. As a result of this process, molybdenum trioxide (MoO_3) and molybdenum dioxide (MoO_2) are formed; this significantly reduces the tribological properties of the film because oxide films have a higher coefficient of friction. The stronger the oxidation of molybdenum, the higher the temperature at which it occurs. The temperature at which the oxidation rate of molybdenum disulfide films increases dramatically depends on how the surface is prepared before coating, how it is applied, and the method used to apply the coating. Coatings applied mechanically, for example, by painting, as a rule, have a higher temperature at which intense oxidation begins compared to deposited coatings. This may be because the particles in suspended coatings are larger, which prevents them from oxidizing too quickly, or because the deposited coating particles are more porous, which accelerates the oxidation process. The structure of the molybdenum disulfide coating also affects its resistance to oxidation. A crystalline structure resists oxidation better than an amorphous one [7]. Meng et al. [11] examined the effectiveness of a MoS_2 coating applied to GCr15 steel under dry sliding conditions at high temperatures. They investigated the effect of different temperatures, loads, and rotational speeds on the properties of the coating. The researchers found that the transition temperature was 350 °C, above which the coefficient of friction and wear of the counter body increased. The authors attribute this to the formation of MoO_3 with increasing temperature. McMurtrey et al. [12] presented research on the development of a high-temperature coating for a petal sliding bearing in an engine turbine. The coating had to withstand short-term cyclic temperature increases up to 810 °C. The authors proposed a composite multilayer coating based on the coatings of Korolon 1350A with a top layer of Korolon 800. The tests included 500 start–stop cycles at temperatures up to 810 °C. Afterward, the bearing was installed in a turbojet engine with a thrust of 240 pounds and a rotation speed of 54.000 revolutions per minute. The engine operated more than 70 start–stop cycles for 14 h. However, neither the composition of the binders used for the coating nor the composition of the solid lubricant base was specified in the work.

The requirements for the maximum operating temperature for friction units are constantly increasing. Technologists are offering new basic solid lubricants, additives, and application technologies to meet these requirements. In the last decade, various other options for solid lubricants have been proposed, especially for aerospace applications. These include niobium diselenide ($NbSe_2$), niobium disulfide (NbS_2), niobium ditelluride ($NbTe_2$), hexagonal boron nitride (h-BN), titanium dioxide (TiO_2), lead oxide (PbO), cerium fluoride (CeF_3), barium difluoride (BaF_2), and calcium difluoride (CaF_2) in various configurations.

For friction and wear tests of new combinations of materials, it is necessary to conduct experimental studies under conditions close to operating conditions.

The goal of this study is to investigate the antifriction properties of suspension solid-lubricating coatings based on molybdenum disulfide at high temperatures. The study examined how these properties change depending on the type of substrate, binder, additives, and load parameters. Different types of solid lubricants were used for comparison.

2. Materials and Methods

In this study, various combinations of molybdenum disulfide with different additives and binders were used (Table 1). The MoS_2 coatings were applied using suspension technology. Before applying solid lubricants to the discs, the substrate surface was treated with a solvent and then sandblasted. After sandblasting, the substrate surface was blown with compressed air and then flushed with a special cleaner–activator (Modengy, Bryansk, Russia). The surface roughness of the disc before coating was 1 μm Ra. In order to compare the samples under study, three different coatings based on graphite, polytetrafluoroethylene, and tungsten disulfide were also investigated. All coatings were cured in air at the temperature recommended by the manufacturer. The thickness of the coatings was 15–20 microns. As can be seen in Table 1, all coatings used different types of binders.

Table 1. Coating samples for testing.

Group	Sample Designation	Range of the Working Temperature, °C	Substrate Material	Binder	Solid Lubricant	Additives
1	1001	250–440	AISI 430	Titanate	Molybdenum disulfide	Graphite
	1002			Organic thermoplastic polymer	Molybdenum disulfide	Sb_2O_3
	1003			Formaldehyde	Molybdenum disulfide	Graphite
	1005			Epoxy	Molybdenum disulfide	TiC
	1006			Polyamide-imide	Molybdenum disulfide	Graphite, WS_2
	1007			Polyamide-imide	Graphite	-
	1066			Polyamide-imide	Molybdenum disulfide	Graphite
	1009			Epoxy	Polytetrafluoroethylene	-
2	1001-01	440–730	Inconel X-750	Titanate	Molybdenum disulfide	Graphite
	1054			Inorganic	Graphite	-
	1055			Inorganic	Graphite	Ceramics
	2560			Organosilicon	Tungsten disulfide	Graphite

The samples in Table 1 were divided into 2 groups depending on their heat resistance. The first group included coatings with a declared heat resistance in the range from 250 °C to 440 °C and the second in the range from 440 °C to 730 °C. The first group includes samples that were deposited on a substrate made of AISI 430 stainless steel, and the second group includes samples that were deposited on a substrate made of Inconel X-750 high-temperature alloy. The binders used are mainly temperature-resistant inorganic compounds such as silicates, phosphates, and titanates. Only sample 2560 used an organic high-temperature binder (organosilicon), which can withstand temperatures up to +500 °C.

Some coatings on the samples contain additives that can improve their performance; for instance, the coatings of samples 1002, 1005, and 1006 contained additives based on antimony, titanium, and tungsten, respectively. The chemical composition of these samples is shown in Table 2.

Table 2. Chemical composition of the coatings of the studied samples 1002, 1005, and 1006.

Sample Designation	C	O	Si	S	Ti	Mo	Sb	W
1002	34.9	16.1	0.4	8.4	-	13.9	26.2	-
1005	60.2	15.7	0.2	8.4	3.1	11.7	-	-
1006	44.1	13.5	1.6	11.3	-	16.4	-	13.1

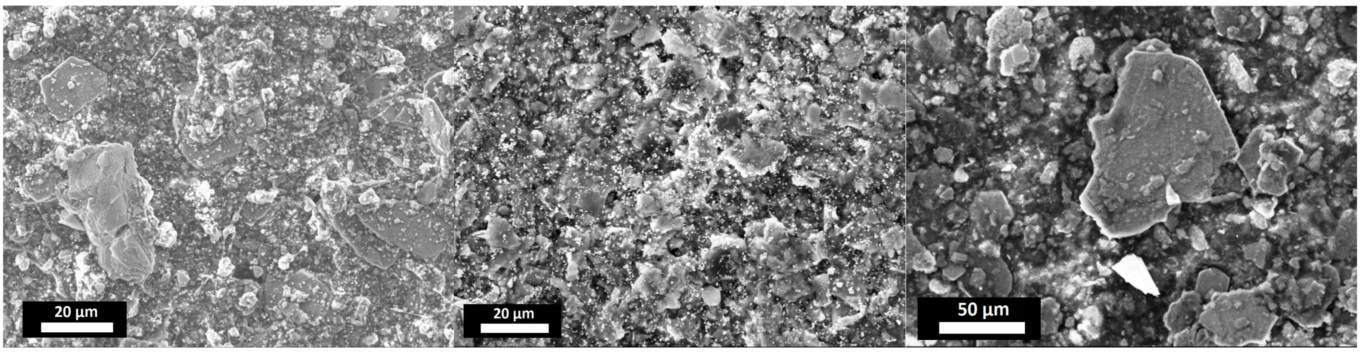
The surface morphology of coatings was studied using the scanning electron microscope (SEM) VEGA 3 LMH (Tescan, Brno, Czech Republic) equipped with an energy-dispersive X-ray spectrometer (EDS).

Friction tests were carried out according to the ball–disc scheme, in accordance with the ASTM G99 standard on a DUCOM POD 4.0 tribometer (Ducom Instruments, Bangalore, India). This scheme is used to compare the antifriction properties of materials and coatings based on their coefficients of friction. In these tests, a silicon nitride (Si_3N_4) ball with a diameter of 6 mm was used as the counter body. The tests were performed with fixed loads (10 N and 23 N) and a linear velocity of 0.1 m/s under gradual heating up to 800 °C in an air environment. During the test, the temperature was gradually increased at a rate of 20 °C/min from ambient temperature, and the test was stopped when the coefficient of friction (COF) reached a value greater than or equal to 0.3. COF = 0.3 is considered the maximum operating value for MoS_2-based coatings [13] and was corroborated in other studies [14–17] in which it was reported that the failure of the lubricating coating occurred at this value. A comparison of the COFs of the coatings was carried out at 150 °C, a temperature at which complete evaporation of moisture from the coating is ensured, and the failure of the coating under the influence of temperature had not been reached.

In this study, the temperature at which the COF of the coating reached a value greater than or equal to 0.3 was used as the temperature resistance criterion.

3. Results

Figure 1 shows the SEM images of sprayed coatings based on molybdenum disulfide particles distributed over different binders (samples 1002, 1006, and 1005). As can be seen, the particle sizes of MoS_2 powder vary within the range of 5–100 microns.



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

Figure 1. The SEM images of coatings: (**a**) sample 1002 (magnification ×1000), (**b**) sample 1006 (×1000), (**c**) sample 1005 (×500).

Figure 2 shows the distribution of sulfur from the MoS_2 compound on the surface of sample 1002, which indicates the homogeneous distribution of MoS_2 in the coating.

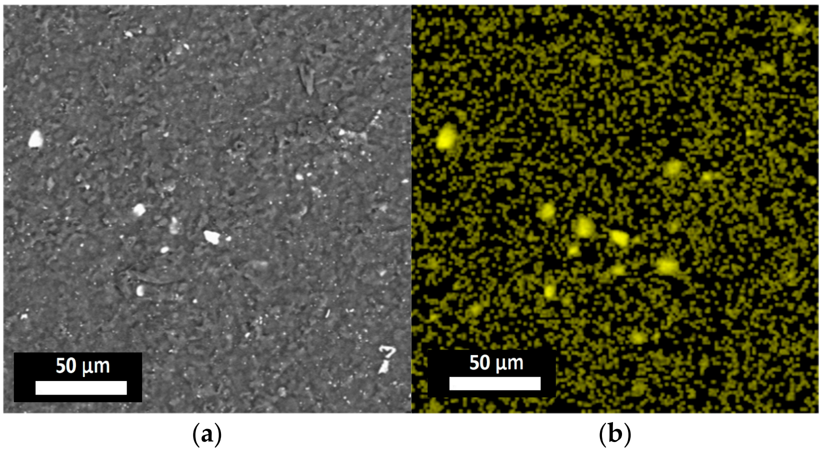


Figure 2. SEM image of the microstructure (**a**) and elemental mapping of sulfur (**b**) from the MoS_2 sprayed on sample 1002.

Figures 3 and 4 show the results of measuring the coefficient of friction at 150 °C. For molybdenum disulfide, this temperature can be considered optimal. At this temperature, any moisture contained within the pores of the coating is completely removed, thereby preventing its influence on the coating properties.

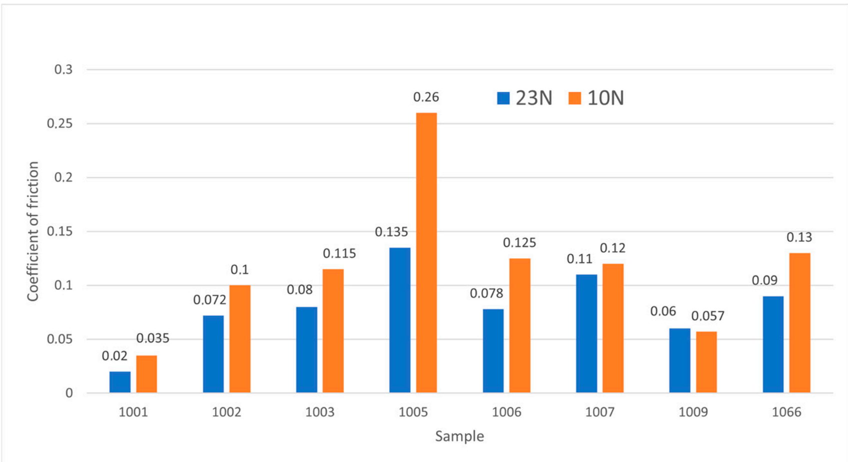


Figure 3. Results of measuring the coefficient of friction of coatings deposited on AISI 430 steel (group 1) at 150 °C.

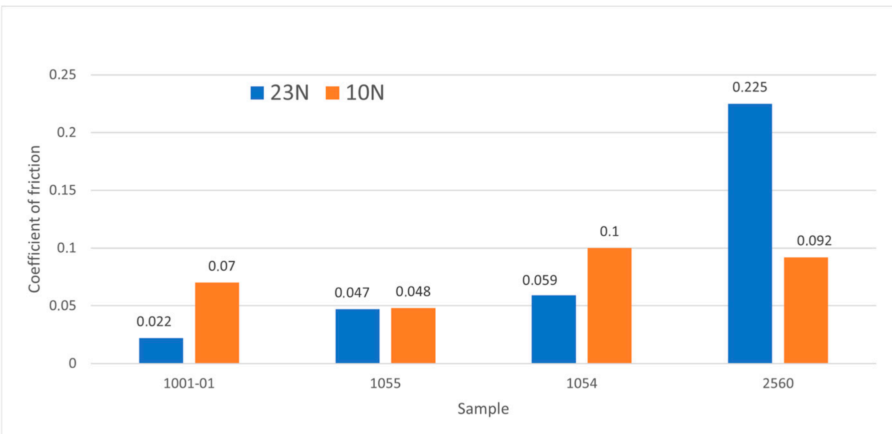


Figure 4. Results of measuring the coefficient of friction of coatings deposited on Inconel X-750 steel (group 2) at 150 °C.

As can be seen in Figure 3, a decrease in the coefficient of friction with increasing load can be considered a general pattern for coatings sprayed on the AISI 430 substrate (group 1). The reduction in COF for samples 1001, 1002, 1003, 1005, 1006, and 1066 ranged from 28 to 48%. On the other hand, samples 1007 and 1009 showed a slight difference in the COF. Among the samples in group 1, the coating of sample 1001, which contains titanate, MoS_2, and graphite (as binder, solid lubricant, and additive, respectively) showed the lowest coefficient of friction at 150 °C. In the same way, the decrease in the COF can be considered a general pattern for coatings sprayed on the Inconel 750-X substrate when the load increases from 10 to 23 N (Figure 4).

The reduction in COF for samples 1054 and 1001-01 ranged from 41 to 68.6%. Sample 1055 showed an insignificant difference in the coefficient of friction, decreasing by only 2%; however, sample 2560 showed an abnormal increase of almost 2.5 times. The lowest coefficient of friction at a temperature of 150 °C was observed in sample 1001-01, which used titanate, MoS_2, and graphite as the binder, solid lubricant, and additive, respectively, for coating.

Another part of this work was the determination of the heat resistance of each coating from groups 1 and 2. Table 3 shows the critical temperature of studied coatings at different loads, in which the COF reached a value of 0.3. The highest critical temperature was observed for sample 1054, which demonstrated performance up to 564 °C under a load of 10 N and 624 °C under a load of 23 N.

Table 3. Heat resistance of the coatings of the samples.

Sample Designation	Temperature at μ = 0.3 (Load 10 N), °C	Temperature at μ = 0.3 (Load 23 N), °C
1001	**477**	**480**
1002	152	431
1003	504	509
1005	435	420
1006	355	418
1007	477	494
1066	456	452
1009	352	407
1001-01	**483**	**496**
1054	564	624
1055	554	562
2560	471	171

From Figures 3 and 4, it can be noted that the coatings of samples 1001 and 1001-01 showed the lowest coefficients of friction at 23 N, and these samples were selected for further study. Figure 5 shows a graph containing two curves that indicate the change in the coefficient of friction curves for samples 1001 and 1001-1 obtained under a load of 23 N. Moreover, this figure also displays the temperature change curve during friction tests.

The coating of sample 1001, with the AISI 430 stainless steel substrate, exhibited a rapid break-in and a reduction in the COF to a minimum value of 0.02, while the temperature of the sample was in the range of 60–80 °C. Then, with a further increase in temperature, there was a gradual increase in the COF to a value of 0.07 when the temperature was around 250 °C. At a temperature of 400 °C, a sharp jump up to a value of 0.3 could be seen, which indicates complete failure of the coating. Moreover, in this sample, a COF below 0.1 can be maintained up to temperatures of 400 °C (Figure 5).

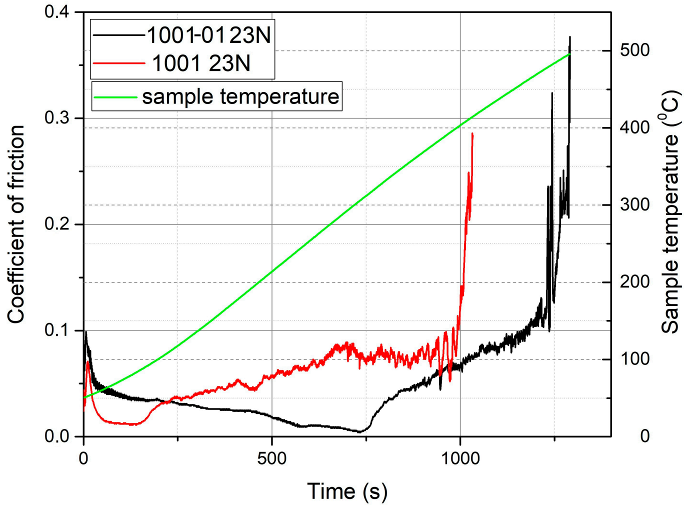


Figure 5. Changes in the coefficient of friction over time for samples 1001 and 1001-01 at a load of 23 N.

On the other hand, the coating in the Inconel X-750 substrate (sample 1001-01) showed a gradual decrease in the COF while the temperature increased. The lowest coefficient of friction (0.016) was observed at 305 °C. The coefficient of friction increased smoothly, as was the case for sample 1001, and then a sharp jump was observed, indicating the complete failure of the coating at 475 °C. In this sample, a COF below 0.1 can be maintained up to temperatures of 450 °C (Figure 5).

As can be seen in Figure 5, the behavior of both friction coefficient curves is similar, but the main differences are the running-in time and the critical temperature of coating failure. At the same time, the coating on the high-temperature alloy substrate showed better performance characteristics.

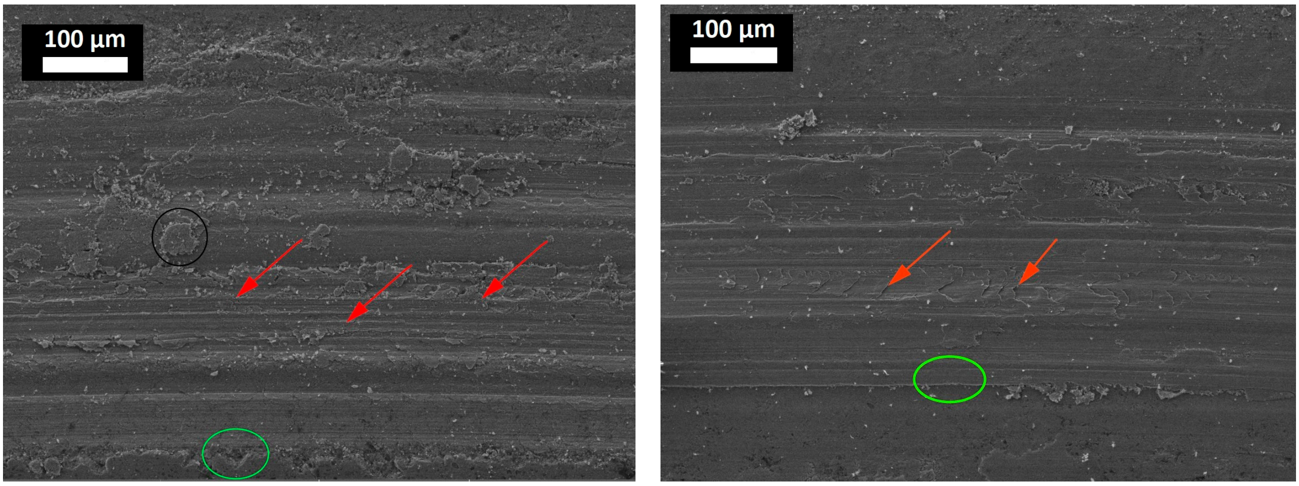
4. Discussion

The obtained values of the coefficient of friction at 150 °C showed that the lowest values were observed for coatings of samples 1001 and 1001-1 containing, in addition to MoS_2, graphite and titanate (Figures 3 and 4). Further research on these two coatings showed that a downward trend was observed until 305 °C in the sample with the Inconel X-750 substrate.

The use of a high-temperature alloy as a substrate can lead to an increase in the critical operating temperature of the coating that contains MoS_2, graphite, and titanate. For example, the operating temperature rises from 477 °C to 483 °C at a load of 10 N, while the operating temperature rises from 480 °C to 496 °C at a load of 23 N (Table 3, samples 1001 and 1001-01).

Moreover, Figure 5 shows the tendency to decrease the coefficient of friction with an increase in the contact load (black curve). This effect can be explained by the phenomenon of coating compaction and a decrease in the shear force in the sliding plane of molybdenum disulfide.

Figure 6 shows the SEM images of the coating surfaces of samples 1001 and 1001-01 after the tribological test. In this figure, it may seem that the two surfaces show a layered structure of the solid-lubricating material. In addition, it is clearly visible that after the friction test, the coating structures are compacted, but the coating of sample 1001-01 is more compacted than that of sample 1001.



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

Figure 6. Surface of sample 1001 after testing under 10 N (**a**) and 23 N (**b**).

Figure 6a shows the surface of sample 1001, where the presence of cracks perpendicular to the sliding direction is noticeable (marked with red arrows), which indicates the brittle nature of the coating failure. In addition, this figure shows that during friction, the coating can be separated in the form of flakes (marked in black), and the boundary of the friction track and the unworn surface has a brittle nature (the zone is marked in green).

Figure 6b shows the friction surface of sample 1001-01, where the presence of layered failure of the solid lubricant is noticeable. The boundaries of the layered failures are indicated in this figure by red arrows. Unlike sample 1001, the interface of the worn track with the unworn surface of the coating of sample 1001-01 appears without traces of brittle failure (the zone is marked in green), which indirectly indicates good adhesion of the coating.

The tendency to decrease the coefficient of friction with an increase in the contact load can be observed in the other samples at 150 °C (Figures 3 and 4), except for sample 2560, and these are inconsistent with the data from the work by Tillmann et al. [8], who also noted a minimum coefficient of friction in the temperature range of 300–375 °C.

The coating of sample 2560 contains tungsten disulfide, graphite, and organosilicon binder sprayed onto the Inconel X-750 substrate and had the maximum coefficient of friction among all samples at 150 °C under a load of 23 N. It can be assumed that such a significant increase in the COF is associated with the antifriction properties of tungsten disulfide, which are lower compared to molybdenum disulfide.

The high-temperature properties of suspension coatings can also be determined by the resistance of the binder material to maintain its properties and integrity at elevated temperatures.

The coatings of samples 1054 and 1055 are based on graphite and binders that should provide short-term performance up to 564 °C and 554 °C (Table 3) under a load of 10 N. Under a load of 23 N, sample 1054 shows a temperature resistance of 624 °C, while the temperature resistance of sample 1055 decreases to 562 °C and is approximately at the same level as with a load of 10 N. This effect can be explained by the presence of ceramic additives in sample 1055 since this is the only difference between them. Similarly, the presence of ceramic additives can explain the fact that the COF at 150 °C and under loads of 10 N and 23 N are at the same level (Figure 7), being the lowest compared to sample 1054 (Figure 4).

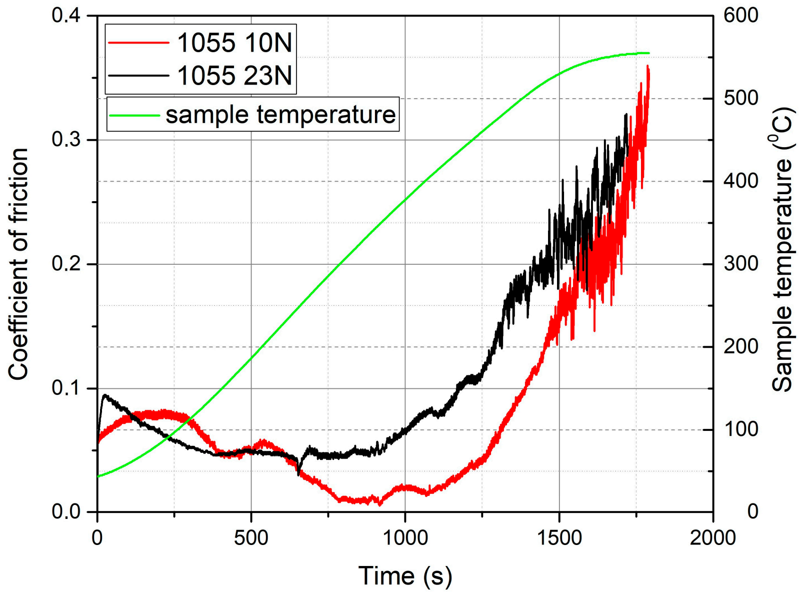
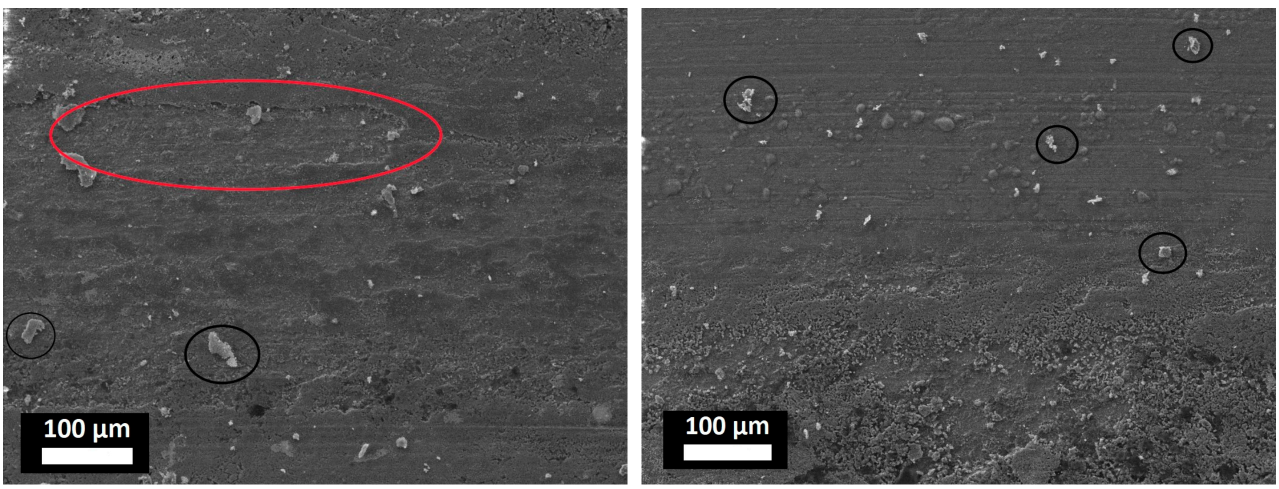


Figure 7. The change in the coefficient of friction over time for sample 1055 at loads of 10 N and 23 N.

Figure 7 shows the behavior of COF over time for sample 1055 at loads of 10 N and 23 N. Here, it can be seen that this sample under a load of 10 N can provide a COF below 0.1 up to temperatures of 500 °C, while this level of COF under a load of 23 N can be provided up to temperatures close to 430 °C. This figure clearly shows that the coefficient of friction of graphite-based coatings gradually increases as they lose their antifriction properties due to their failure (see the curve behavior change after 1350 s and 1500 s for the black and red curves, respectively). This observation is contrary to the behavior of the COF of molybdenum disulfide, which increases sharply when it loses its lubricating properties (see in Figure 5 the curve change after 1000 s and 1250 for the red and black curves, respectively).

Figure 8 shows the SEM images of the coating surfaces of sample 1055 after testing under 10 N and 23 N. From Figure 8a, it is possible to conclude that during testing under 10 N, large coating particles peel off. The place where this peeling occurs is shown in Figure 8a and is highlighted in red. In addition, in this figure, one can see how small particles of the coating (highlighted in black) that peeled off during the friction process are found on the friction track.



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

Figure 8. Surface of sample 1055 after testing under 10 N (**a**) and 23 N (**b**).

Unlike the surface of sample 1055 at 10 N, no large-sized particles of the coating that peeled off from the surface were observed after testing under 23 N. In addition, it is clearly visible that this surface appears dense, with traces of small coating particles highlighted in black (Figure 8b).

5. Conclusions

The study of the antifriction properties of suspension solid-lubricating coatings based on molybdenum disulfide at high temperatures, depending on the type of substrate, binder, additives, and load parameters, was carried out. The solid-lubricating coatings used in this work were compounds based on MoS_2 with different additives (graphite, Sb_2O_3, TiC, WS_2) and binders (organic and inorganic in nature). These coatings were compared with coatings based on graphite, polytetrafluoroethylene, and tungsten disulfide. Moreover, the solid lubricants were sprayed on two different substrates (Inconel X-750 high-temperature alloy and AISI 430 stainless steel) and tested in accordance with ASTM G 99 under loads of 10 N and 23 N at temperatures from 25 °C to 800 °C in an air environment.

Based on the results obtained, the following conclusions can be drawn:

(1) The antifriction properties of solid lubricant suspension coatings at high temperatures significantly depend on the type of solid lubricant material and the binder used. The coatings containing MoS_2, graphite, and titanate, as solid lubricant, additive, and binder, respectively, showed the lowest coefficient of friction at 150 °C under 23 N.

(2) The tendency of the coefficient of friction to decrease with increasing contact load for coatings based on MoS_2 and graphite was established. The coating containing graphite, ceramic, and an inorganic binder applied to an Inconel X-750 alloy substrate can maintain a COF below 0.1 up to temperatures of 500 °C when tested under a 10 N load while testing this coating under a 23 N load shows that a COF below 0.1 can be maintained up to temperatures of about 430 °C.

(3) The use of Inconel X-750 as a substrate can lead to an increase in the critical operating temperature of coatings that contain MoS_2, graphite, and titanate. For instance, at a load of 10 N, the operating temperature increased from 477 °C to 483 °C, while at a load of 23 N, the operating temperature increased from 480 °C to 496 °C.

(4) The coating containing MoS_2, graphite, and titanate can maintain a coefficient of friction below 0.1 up to temperatures of about 400 °C or 450 °C when applied on AISI 430 stainless steel or Inconel X-750 alloy substrates, respectively.

(5) The coating containing graphite as a solid lubricant, ceramics as an additive, and inorganic binder showed the best performance in terms of a combination of properties (low coefficient of friction and longer operation, with a coefficient of friction below 0.3 under increasing temperature) when it was applied on the X-750 Inconel alloy substrate.

(6) It was established that the coefficient of friction of graphite-based coatings gradually increases as they lose their antifriction properties due to their failure, while the coatings based on molybdenum disulfide show the opposite behavior, where the coefficient of friction increases sharply when it loses its lubricating properties.

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Conflicts of Interest: Author Maksim I. Prudnikov was employed by the company LLC Modengy. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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