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Structure and properties of AlB_{12}-Al detonation coatings on steel 45

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The article is devoted to the study of the properties of detonation coatings AlB_{12}-30 wt.% Al (AB70) and AlB_{12}-70 wt.% Al (AB30) applied to 45 Steel using the DNEPR-5M installation. The coatings have the following characteristics: thickness h ~ 170-200 μm, $H_μ$ = 27-29 GPa, HV = 4.35 GPa. Changes in the tribological characteristics of the composites depending on the friction mode are analyzed. The AB70 composition was found to have stable tribological characteristics: f = 0.21-0.38, wear rate I = 3.3-8.6 μm/km. Coated samples outperform uncoated 45 steel in wear resistance. Changes in the surface structure after friction are shown depending on the sliding speed and load. Two wear mechanisms have been identified: oxidative and adhesive-abrasive. It was concluded that it is possible to use the AlB_{12}-Al system for applying detonation coatings.

Keywords: AlB_{12}-Al, detonation coatings, microstructure, hardness, tribological characteristics

Структура та властивості детонаційних покриттів системи AlB_{12}-Al. *О.П. Уманський, В.Б. Муратов, В.Є. Шелудько, К.М. Гальцов, О.А. Бондаренко, Т.В. Мосіна, В.Л. Сироватка, І.С. Марценюк, В.Т. Варченко, В.В. Кременицький, А.В. Міницький, О.В. Кущев*.

Статтю присвячено дослідженню властивостей детонаційних покриттів AlB_{12}-30 мас.% Al (АВ70) та AlB_{12}-70 мас.% Al (АВ30), нанесених на Сталь 45 на установці "ДНІПРО-5М". Покриття мають такі характеристики: товщина h ~ 170-200 мкм, $H_μ$ = 27-29 ГПа, HV = 3.67-4.35 ГПа. Проаналізовано зміну трибологічних характеристик композитів в залежності від режиму тертя. Встановлено, що покриття АВ70 має стабільніші трибологічні характеристики: f = 0.21-0.38, інтенсивність зношування I = 3.3-8.6 мкм/км. Зразки з покриттями перевищують сталь 45 без покриття за зносостійкістю. Показано зміну структури поверхні після тертя в залежності від швидкості ковзання та навантаження. Встановлено прояв двох механізмів зношування – оксидного та адгезійно-абразивного. Зроблено висновок щодо можливості застосування системи AlB_{12}-Al для нанесення детонаційних покриттів.

1. Introduction

The properties of the working surfaces of parts and structural elements of machines and constructions are determining factors in their operational reliability and service life [1]. Increasing the service life of machine parts can be achieved by applying coatings on their surfaces protecting them from corrosion and erosion, wear, high-temperature action of aggressive environments, etc. [2-4].

In this regard, detonation spraying (D-spraying) has become widespread. This method is based on the principle of heating the sprayed material (usually a powder) followed by its acceleration and transfer to the sprayed part using the detonation products of gas-oxygen fuel (usually mixtures of propane and butane or acetylene and oxygen are used) [5]. Due to the high velocity of the sprayed particles (600-1000 m/s), D-coatings have minimal porosity (~1%) and significant adhesive strength (40-200 MPa) of the coating to the part, depending on the sprayed material. Moreover, heating of the sprayed product is insignificant (up to ~250 °C), which helps prevent volumetric changes in the part. In addition, detonation spraying is a very economical, but not very productive method (compared to, for example, high-velocity thermal spraying). As a rule, it is most economical to spray the product on a surface area of no more than a few square centimeters.

D-spraying allows for the application of a wide range of materials: metals and alloys, oxides, their mixtures, hard metal-ceramic alloys based on tungsten carbides, chromium, titanium, mixtures of carbides with metals, as well as borides [1, 5, 6-10].

Among the latter, aluminum dodecaboride AlB_{12} is of interest. The method of its synthesis from Al and BN developed at the Frantsevich Institute for Problems of Materials Science of NAS of Ukraine, turned out to be more rational and cost-effective than direct synthesis from Al and B [11]. AlB_{12} has a low density (~2.52 g/cm^3), and the features of its crystalline structure (icosahedral boron framework) determine its high hardness (22-24 GPa), and fusibility (2070 °C) [12].

However, the low crack resistance of aluminum dodecaboride significantly limits the scope of its application [12]; therefore, it is advisable to use AlB_{12} in combination with ductile metal. It is promising to use aluminum, which is characterized by high plasticity, low density (2.7 g/cm^3) and low melting point (660 °C). It was previously established that Al wets AlB_{12} well, with the formation of contact angles ($\Theta \approx 20$ deg), and there are no secondary phases in the interaction zone at the Al-AlB_{12} interface [12]. In particular, the composite material AlB_{12}-50 wt.% Al was used in the deposition of electric spark coatings on titanium (VT1-0) and aluminum (D1) alloys, as well as on 45 Steel [13-15]. It should be noted that there is no information in the literature on the use of the AlB_{12}-Al system for applying D-coatings.

So, the aim of the work is to investigate the possibility of using the composite powder material "aluminum dodecaboride AlB_{12}-Al" to obtain D-coatings on 45 Steel, and to investigate the structure and properties of these coatings.

2. Experimental

Powder materials used in this study were high-purity Al (99.8 %) and AlB_{12} produced by CVD method due to reaction of hexagonal boron nitride and aluminum [16]:

$$12BN + 13Al \rightarrow AlB_{12} + 12AlN$$

The average size of AlB_{12} powder particles was 150÷200 nm. The AlB_{12}-Al composite powder was produced by conglomerating the starting components in an organic binder (zapon) followed by sieving to obtain particles in the size range of 63...20 μm. In such a way, two compositions AlB_{12}-30 wt.% Al and AlB_{12}-70wt.% Al hereinafter referred to as AB70 and AB30, respectively, were obtained.

D-coatings were applied to a 1×1 cm steel 45 (GOST 1050-88) substrate using a DNEPR-5M installation in the following mode: spraying distance L = 170 mm, combustible mixture: acetylene C_2H_2/oxygen O_2 = 20/30 g, frequency f = 4 Hz, spraying time t = 10 s.

The microhardness of the D-coatings was measured using a PMT-3 microhardness tester at a load of P = 0.5 N. The Vickers hardness HV was measured using a FALCON500 stationary hardness tester (INNOVATEST, Netherlands) at a load of P = 1.96 H.

Tribological properties of the D-coatings were studied on an M-22M friction machine in accordance with GOST 26614-85 under the following conditions: P = 2, 4, and 6 MPa, V = 2 and 4 m/s and friction path S = 2 km. Hardened 45 Steel (HRC 45-50) was used as the counterbody.

X-ray diffraction analysis of the D-coating surface was performed on a Rigaku Ultima-IV

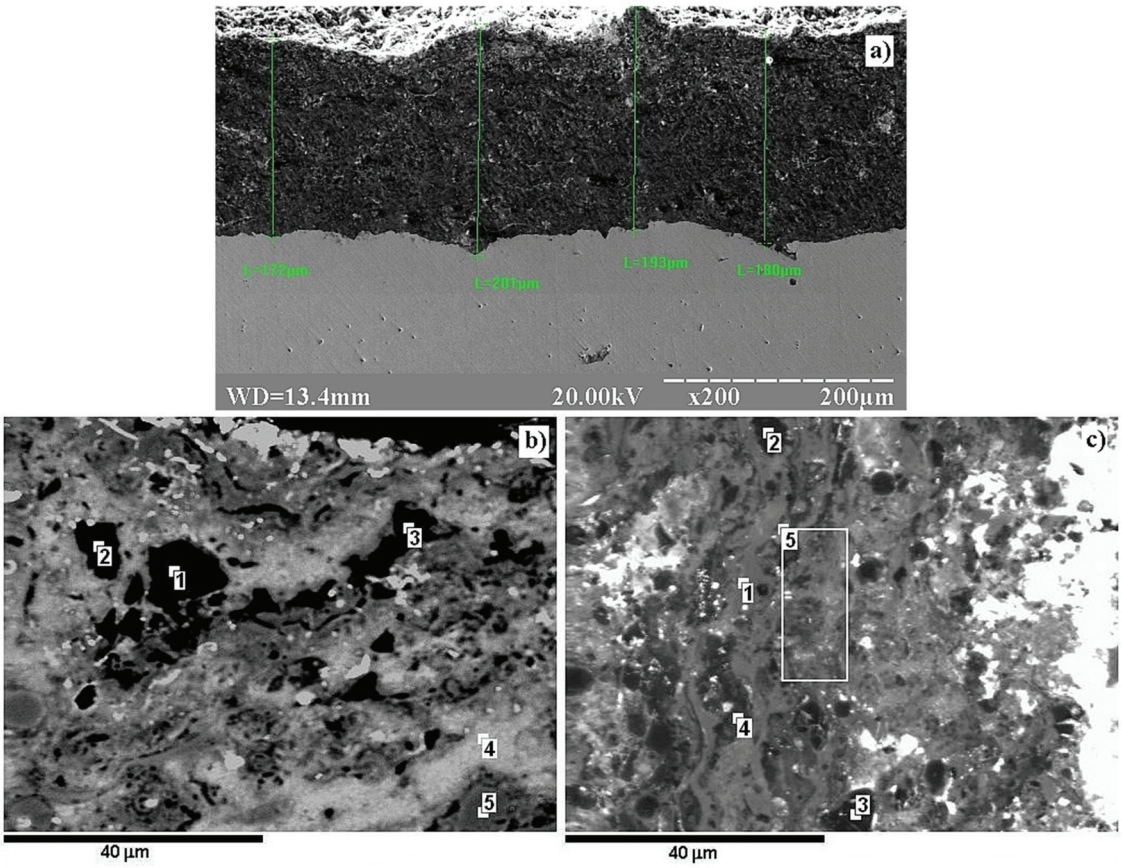

Fig. 1 – Microstructure of cross-section of D-coatings with the results of local elemental analysis: a – general view (by the example of AB70 coating); b – AB30 D-coating; c – AB70 D-coating

diffractometer (Rigaku Corp., Tokyo, Japan) using CuK_α-filtered radiation (λ = 1.54051 Å).

The microstructure of the D-coatings was studied using a JEOL JSM-6490 LV SEM equipped with INKA Energy 350XT energy-dispersive spectrometer (Oxford Instruments plc.) and a REM-106I SEM (JSC "SELMI", Sumy, Ukraine).

3. Results and discussion

The general view and microstructure of the AB70 and AB30 D-coatings cross-section are shown in Fig. 1. The average thickness is 170-200 µm, and the microhardness is H_μ = 27-29 GPa. However, there are also sections with H_μ = 7-9 GPa and 0.9-2.8 GPa. The Vickers hardness, measured perpendicular and parallel to the coating thickness, averages HV = 4.35 and 3.67 GPa for the samples AB70 and AB30 respectively.

The microstructure of the AB30 composite is characterized by the presence of large aggregates and linear formations (lamellae) of dark-colored AlB_{12} with light-colored zones of aluminum oxide between them. The large amount of ductile aluminum in the AB30 composite increases the viscosity of the deposited compound and retains large AlB_{12} aggregates, which contribute to abrasive wear during friction by crushing or shearing protrusions on the softer counterbody. Unlike the AB30 composite, the surface of the AB70 one is more uniform. AlB_{12} particles, 2.9-3.4 µm in size, are more evenly distributed within the Al_2O_3 matrix. On the one hand, these particles adhere more tightly to the counterbody surface, increasing the tribocontact area; on the other hand, they also promoting more intense oxidation with the formation of oxide layers. The periodic destruction of these layers reduces the strength of the microvolumes in the surface layer of the tribocontact, promoting oxidative wear.

The results of the local elemental analysis of the AB70 and AB30 D-coatings and their phase compositions are presented in Table 1 and Fig. 2. The data in Table 1 indicate that both composites contain regions with increased Al ((~80 wt.% and ~57 wt.%) and oxygen (~47 wt.% and ~43 wt.%) content for AB30 and AB70 respectively. Boron is part of AlB_{12}, and

Table 1 – Results of local elemental analysis of AB30 and AB70 D-coatings

Spectrum	Element, wt.%				Total
	B	O	Al	C	
AB30 D-coating					
1	82.91		17.09		100
2	83.16		16.84		
3	81.67	1.27	17.06		
4		4.27	79.09	16.64	
5		46.92	53.08		
AB70 D-coating					
1		43.16	50.69	6.15	100
2	83.09		16.91		
3	81.87	0.61	17.52		
4	68.99	10.77	20.24		
5		32.89	57.11	8.67	

oxygen and aluminum form secondary structures (mainly oxides) during friction.

Elemental analysis (Table 1) confirms the presence of elements corresponding to the main phases (Al, Al_2O_3, AlB_{12}, Fig. 2) in the coatings.

Fig. 3 shows the results of tribological tests of AB30 and AB70 samples in dry friction mode. These data indicate that with increasing load and sliding speed, the friction coefficient decreases and wear intensity increases. Compared to uncoated 45 Steel, the AB30 and AB70 composition exhibit significantly improved wear resistance. With increasing load up to 6 MPa, the wear of uncoated 45 Steel becomes catastrophic.

As noted above, the AB30 composite is characterized by the presence of large AlB_{12} aggregates affecting abrasive wear during friction. Moreover, with increasing load, a sharp increase in the wear rate of the friction pair is observed. At the same time, the friction coefficient decreases slightly from 0.44 to 0.3 (V = 2 m/s), which may be due to the destruction of the formed aggregates of the secondary structure (oxide layer). At the same time, the friction coefficient also decreases with increasing sliding speed, since the contact time at the points of contact between the specimen and the counterbody decreases. A sharp increase in wear rate should be noted at V = 4 m/s and a load of P = 4 MPa, which may be due to intense (T < 400 °C) oxidation during contact.

Unlike the AB30 sample, the AB70 coating is characterized by more stable tribological properties. Therefore, the AB70 composition was studied in more detail (at the sliding speed V = 4 m/s).

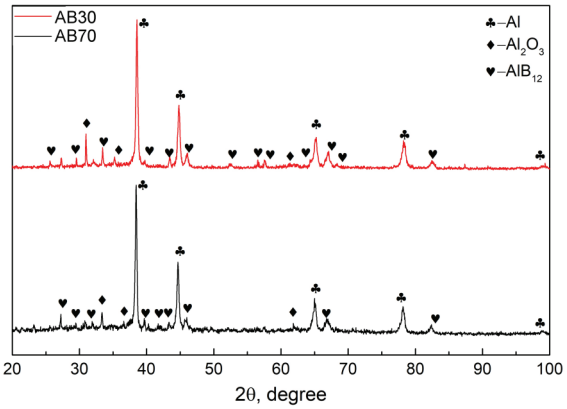

Fig. 2 – Diffraction patterns of AB30 and AB70 D-coatings on 45 Steel

Fig. 4a shows the surface of the AB70 coating after friction on 45 Steel (P = 2 MPa, V = 4 m/s). A characteristic feature is the presence of dark areas consisting of AlB_{12} aggregates penetrated by light particles (ranging in size from 3 to 6.2 μm). The light areas are represented by elongated conglomerates or individual grains. A characteristic feature is the presence of zones with a high oxygen content (up to 51.73 wt.%) and iron (up to 77.23 wt.%), which indicates the formation of Al and Fe oxides during friction (Table 2). Hard AlB_{12} fragments act as an abrasive on the counterbody surface, crushing and cutting off protrusions, facilitating the transfer of iron from the counterbody. Thus, areas with iron inclusions appear on the surface. Heating of the surface (T ~ 230 °C) during friction promotes the oxidation of Al and Fe with the formation of solid oxides which also contribute to abrasive wear. Thus, in this case, two conjugated wear mechanisms are present: oxidative and adhesive-abrasive.

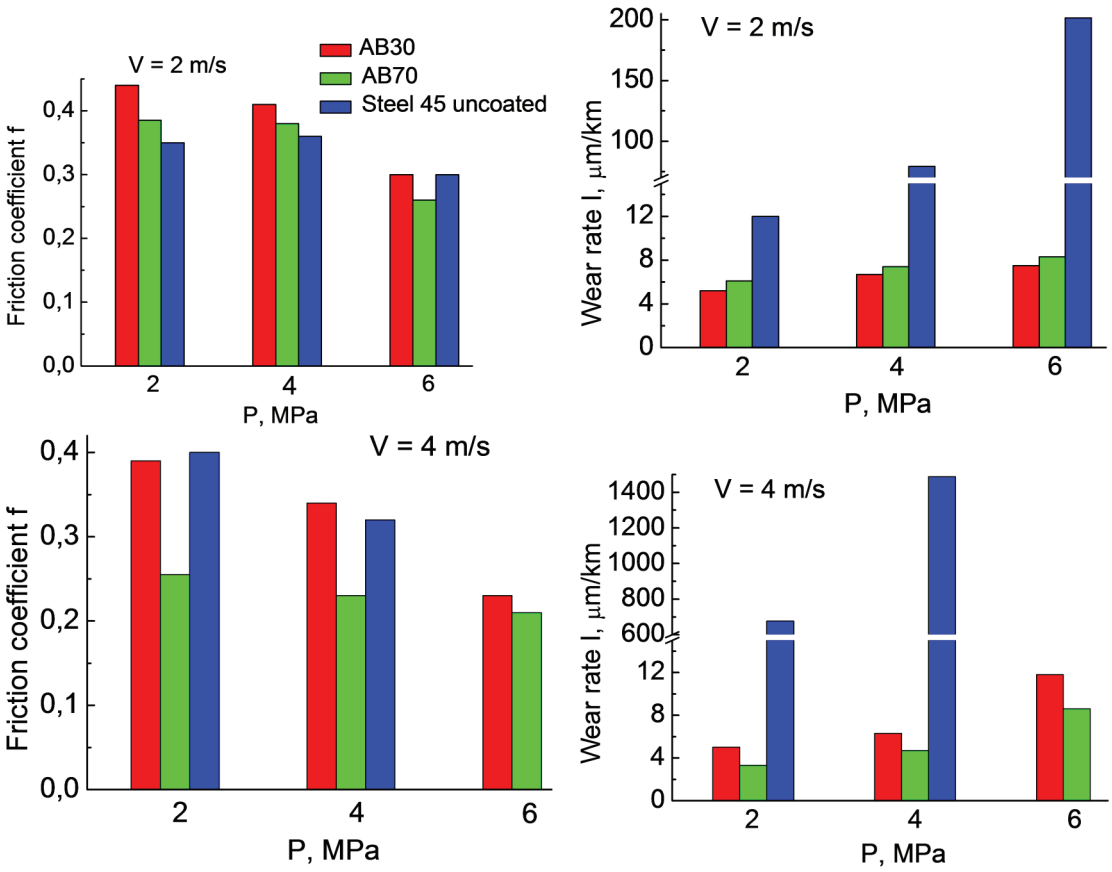

Fig. 3 – Tribological characteristics of AB30 and AB70 D-coatings on 45 Steel and uncoated 45 Steel under different loads and sliding speeds

When the load increases to P = 4 MPa at the same sliding speed, the relief becomes ordered and alternating layers are formed (Fig. 4b). Layers with dark AlB_{12} inclusions (62 to 100 μm in size) alternate with light layers 212 to 225 μm in width. Local microanalysis data (Table 2) indicate that this mode results in the formation of larger AlB_{12} grains, distributed within the layers and between them as fine particles, covering the entire surface. As a result, the AlB_{12} particles exhibit a more intense abrasive effect, promoting the formation of iron in the contact zone, followed by the formation of complex $Al_xFe_yO_z$ oxides.

The system we consider as a frictional contact is dissipative and meets the criteria of self-organization (system openness, nonlinear processes of deformation and wear, formation of secondary structures, etc.). During friction, incoming energy is dissipated into other forms of energy, particularly heat, and accumulated by secondary structures, resulting in self-organization of the system. Furthermore, they implement the principle of structural dissipation of accumulated energy into structural elements in the form of elastic energy [17].

Like most structural materials, the composite under study is statistically heterogeneous, which determines the nonlinearity of the deformation and wear processes during frictional interaction [18]. The authors of [19, 20] note the role of secondary contact structures in the boundary layers of the metal during friction and wear. Such secondary structures can be oxide films in the boundary layers or a supersaturated oxygen solution in plastically deformed surface volumes of the metal, defects and damage of structures in the contact volumes, etc. [21]. During friction, as a result of the interaction of particles of the original disordered system, the deformation contact adapts to changing conditions (temperature, sliding speed, and load). The most important result of such adaptation is the self-organization (ordering) of the system, as a result of which the energy, force, tribomechanical parameters of friction and wear are reduced. These are friction coefficient, temperature, mass wear of the sample and wear intensity [19].

In our system, with a further increase in load to P = 6 MPa, the surface morphology changes

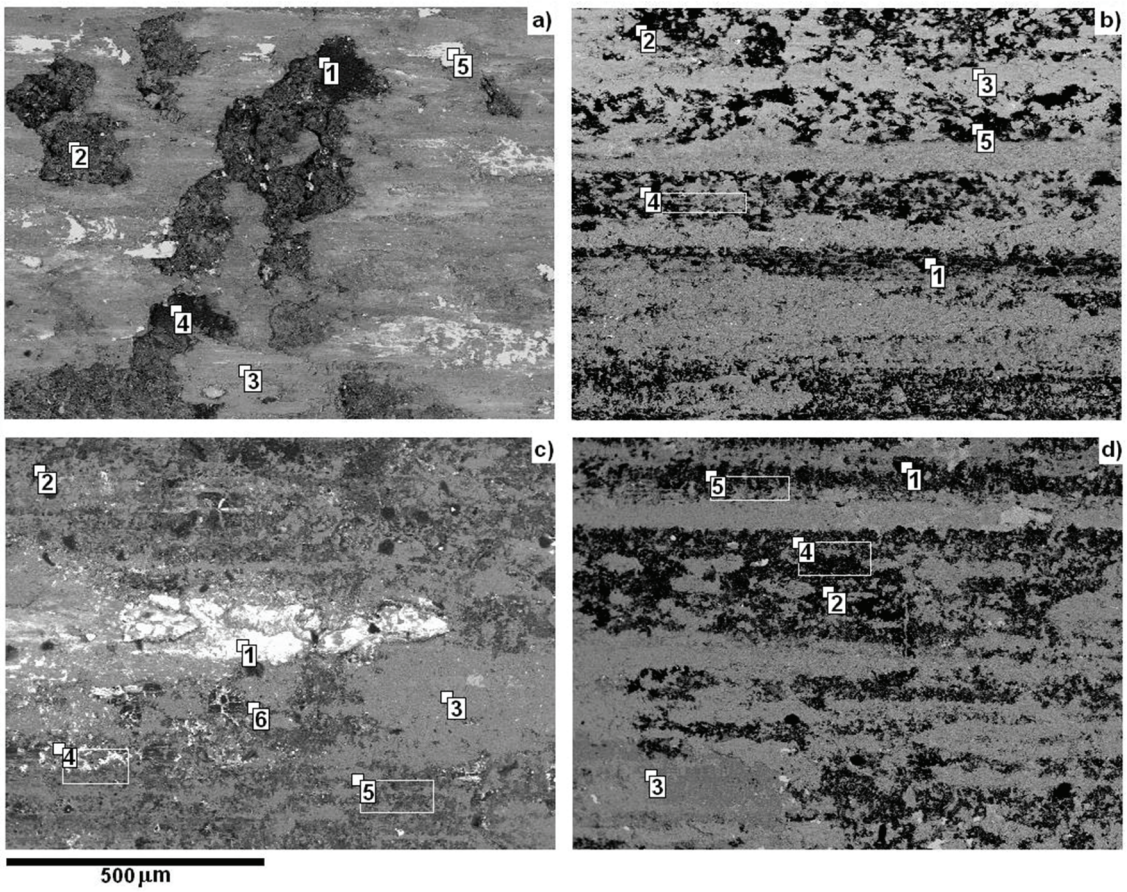

Fig. 4 – Surface of AB70 D-coating under friction on 45 Steel (V = 4 m/s): a – P = 2 MPa; b – P = 4 MPa; c – P = 6 MPa and d – surface of AB70 D-coating under friction on ShKh15 steel (P = 6 MPa, V = 6 m/s) with the results of local elemental analysis

(Fig. 4c). The striped relief structure is transformed into a uniform surface with light gray and darker areas. The continuous AlB_{12} network includes individual dodecaboride grains ranging in size from 12.5 to 25 μm. Note the high iron content at different points, ranging from 4.18 to 79.19 wt.% (Table 2). As a result of surface seizure followed by detachment, a large elongated conglomerate consisting mainly of iron oxide FeO is formed. A more uniform contact surface aids in reducing the friction coefficient to f = 0.21, and the abrasive effect of the resulting oxides increases the wear intensity (Fig. 3).

Any macroscopic system consists of a large number of microscopic states (e.g., volume, temperature, pressure, etc.), characterized by entropy. The evolution of a tribological system consists in achieving a state with entropy lower than its initial value [22]. For example, if the system is heated from temperature T_1 to T_2, the change in entropy ΔS is described by the following expression [23]:

$$\Delta S_{1\to 2} = S_2 - S_1 = \int_{1\to 2} \frac{dQ}{T} \ , \qquad (1)$$

Where $\frac{dQ}{T}$ is the reduced amount of heat imparted to the system during its transition from state 1 to state 2. Taking into account $dQ = mcdT$, where m is the mass of the sample, c is its specific heat capacity, formula (1) will take the form (T_1 = 298 K):

$$\Delta S = mc \ln\left(\frac{T_2}{T_1}\right) \qquad (2)$$

The data in Fig. 3 indicate that the AB70 sample is characterized by minimal values of tribotechnical characteristics compared to uncoated 45 Steel, which is one of the external manifestations of self-organization [19]. At a load of P = 6 MPa and a sliding speed of V = 4 m/s, the result of self-organization of the system is not only a change (ordering) of the surface microgeometry (Fig. 4b, c), but also a decrease in the tribotechnical characteristics. Therefore, it was of interest to compare the change in entropy during friction of a sample with and without a coating. The calculation

Table 2 – Results of local elemental analysis of friction surface of AB70 D-coating (Fig. 4)

Spectrum	Element, wt.%									Total
	B	O	Al	C	Cr	Fe	Si	Mn	Ni	
Fig. 4a										
1	82.67	1.36	15.97							100.00
2		46.06	52.05	0.43	1.11	0.35				
3		51.73	20.93			27.34				
4	82.77	10.45	16.78							
5		21.09	0.93			77.23	0.75			
Fig. 4b										
1	81.49	1.89	16.62							100.00
2	81.72	0.73	17.01			0.54				
3		37.18	10.46			52.36				
4		40.01	28.67			31.32				
5	44.30	26.07	23.98			5.64				
Fig. 4c										
1		22.53	0.46			76.19		0.82		100.00
2	43.90	26.36	25.56			4.18				
3		44.21	18.72	4.44		32.63				
4		30.76	28.55	4.56	1.05	32.47			2.61	
5		45.04	37.35		1.22	16.39				
6	82.02	1.53	16.45							
Fig. 4d										
1	81.07	1.53	15.06						2.34	100.00
2	81.90	1.03	17.07							
3		43.88	15.56	3.79		36.77				
4		41.67	42.70			15.63				
5		40.43	46.07			13.50				

Table 3 – Results of entropy ΔS calculations

Friction mode	Sample temperature T_2, K		Entropy ΔS, J/K	
	45 Steel with D-coating AB70	Uncoated 45 Steel	45 Steel with D-coating AB70	Uncoated 45 Steel
P = 2 MPa V = 4 m/s	500	471	0.00338	0.4654
P = 4 MPa V = 4 m/s	610	496	0.00462	0.5283

was carried out using formula (2) and the results are presented in Table 3. Analysis of the change in ΔS indicates that at V = 4 m/s and P = 2 and 4 MPa, the change in entropy during friction of the AB70 composite is, respectively, 137 and 114 times less, than during friction under the same conditions of uncoated 45 Steel, which confirms the assumption of self-organization of the system during friction.

Analyzing the data in Fig. 4, one can note the following sequence of changes in the tribocontact relief and wear mechanisms depending on the friction mode.

The friction process at P = 2 MPa and V = 4 m/s is characterized by two conjugated wear mechanism: oxidative and adhesive-abrasive.

As the load increases to 4 MPa at the same speed, the microgeometry of the relief changes toward ordering (self-organization). Layers with larger grains of hard AlB_{12} can move over layers with fine particles, reducing the friction coefficient. The composite structure has zones with increased oxygen and iron content due to the formation of oxides through the oxidative wear mechanism. The presence of AlB_{12} aggregates does not exclude the possibility of abrasive wear, leading to the appearance of iron in the contact zone.

With increasing load to 6 MPa, a continuous, uniform relief is formed with dispersed AlB_{12} grains with an increased content of oxygen, aluminum, and iron, as well as particles of the oxide layer (Table 2), indicating an oxidative wear mechanism. In this case, the influence of temperature, which contributes to oxidative wear, cannot be excluded, since in this mode the temperature in the tribocontact zone is ~360 °C.

It is of interest to compare the obtained data with the results of friction in contact with a harder counterbody. Fig. 4d shows the surface of the AB70 coating during friction on ShKh15 ball bearing steel, the hardness of which (HRC61-63) exceeds the hardness of 45 Steel counterbody. The tests were carried out under more severe conditions (P = 6 MPa, V = 6 m/s). The AB70 coating remained resistant to this mode. At friction on a harder counterbody, a less ordered surface structure is formed with wider bands, which mainly contain aluminum, oxygen, and iron. The iron content is lower (from 13.5 to 36.77 wt.%, Table 2), since the harder counterbody is less susceptible to the abrasive action of AlB_{12} and complex oxides. Increasing the hardness of the counterbody makes difficult plastic deformation and microcutting of the friction surface. At the same time, the friction coefficient f = 0.24, the mass wear of the sample increased slightly to 2.8 mg/km, while the wear of the counterbody decreased to 0.18 mg/km. The wear rate of the friction pair is I = 8.2 μm/km. Increasing the temperature in the contact zone to T = 432 °C promotes intensification of the oxidation proceses with the formation of complex oxides $Al_xFe_yO_z$ and the transition to the oxidative wear mode.

4. Conclusions

For the first time, using D-spraying, coatings of the AlB_{12}-Al system with different contents of boride and aluminum components were obtained (AlB_{12}-30 wt.% Al (AB70) and AlB_{12}-70 wt.% Al (AB30)), and some of their properties such as thickness h = 170-200 μm, microhardness H_μ = 27-29 GPa, Vickers hardness HV = 4.35 GPa were determined. The AlB_{12}-Al system can be used to produce detonation coatings on steels.

Tribological tests of the coatings were conducted at sliding speeds V = 2, 4, and 6 m/s and loads P = 2, 4, and 6 MPa. The AB70 composite has the following characteristics: f = 0.21-0.38, I = 3.3-8.6 μm/km.

The structure of the surface layer changes depending on the sliding speed: the alternating strip relief at V = 4 m/s is transformed into a uniform surface at V = 6 m/s, which indicates the processes of ordering (self-organization) of structures in the contact zone with a decrease in the entropy value.

Based on the results of local microanalysis, the formation of iron and aluminum oxides in the friction zone was established. This indicates an oxidative wear mechanism, and the appearance of iron in the contact zone confirms adhesive-abrasive wear as a result of the impact of solid AlB_{12} particles and oxides on the surface of the counterbody.

Replacing the counterbody material with hardened ShKh15 steel made it possible to achieve coating resistance to high friction conditions: f = 0.24, I = 8.2 μm/km. The wear rate of the counterbody has decreased by approximately 35 times (to 1.8 mg/km).

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