



Solid lubricant nanocoating's based on magnesium compounds

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Abstract

The results of studies of friction and wear of detonation composite nanocoating's based on the ternary compound aluminum-magnesium-boron under test conditions with constant load in the sliding velocity field are presented. Their structural-phase composition and passivating complex of surface oxide structures are determined using modern physical analysis methods. It is established that the parameters of wear intensity and friction coefficients are minimized due to the set of surface structures that regenerate a stable self-lubricating structured layer under friction conditions. Factors that influence the formation of dynamic equilibrium of a self-lubricating layer that has an effective ability to self-repair are determined. At the same time, a continuous protective layer screens the processes of molecular-adhesive interaction and blocks the development of unacceptable destruction phenomena.

Keywords: surface structures, carbide graphite, wear, interlayer, phase composition, self-lubrication.

Introduction. As the scope of application of coatings obtained by spraying increases, the requirements for their quality are increasing. Existing methods of improving quality are necessary, but insufficient. The most accessible and most common means of improving operational quality is the use of lubricants. The use of self-lubricating sliding materials, which provide both technical and economic advantages [1, 2], is becoming increasingly important in many industries. The lubricating effect is achieved by incorporating solid lubricants directly into the coating structure or the material matrix itself [3, 4].

Literature review

Considerable attention is devoted to the study of self-healing coatings and the discussion of their self-healing mechanisms and functionality [5]. The authors argue that the use of such coatings is a promising approach to protecting components from wear. The paper [6] analyzes the main problems and current developments in the field of protecting friction surfaces and improving their wear and corrosion resistance. Additionally, it highlights the problems that arise when using self-lubricating coatings. In their works [7, 8], the authors investigated coatings used to protect component surfaces in the automotive industry. Parts used in automobile manufacturing are simultaneously exposed to corrosion and repeated loading and are operated under severe conditions. Therefore, significant attention is devoted to the precise analysis and prediction of the effectiveness of part surface protection.

The use of nanostructured coatings modified with magnesium carbide nanoparticles [9] and the use of solid lubricant coatings [10] deserve, in our opinion, special attention when selecting a method to enhance the wear resistance of materials. Current research by scientists worldwide focuses on methods for producing nanocoatings and using them to protect friction surfaces, particularly those operating under extreme loads and in aggressive environments [11–13]. These studies highlight a comprehensive method for forming protective solid-lubricant layers during friction [14–15]. There is also a number of studies devoted to the analysis of the use of inorganic and polymer nanocoatings, which demonstrate that controlled wear and self-lubrication are key factors in modern mechanical engineering [16–17].

The purpose of the work



Research into the patterns of wear of coatings based on magnesium compounds, study of their structural and phase composition and analysis of the structure formation of antifriction surface layers and their influence on self-lubrication processes.

Materials and research methods

The powders proposed by the authors contain as initial reagents the ternary compound aluminum-magnesium-boron (AlMgB_{14}), which is distinguished by significant mechanical and thermal properties and is used as a base for composite coatings for the first time. In order to improve the tribotechnical characteristics, the base material was additionally doped with silicon (Si), aluminum (Al), nickel (Ni), titanium (Ti), chromium (Cr), zirconium (Zr), carbon (C). The choice of alloying additives is determined by tribomaterials science provisions, which thoroughly combined the obtained results into a single system of ideas about magnesium compounds with high tribotechnical properties. Further obtaining high-quality self-lubricating coatings involved the use of modern technologies, including mechanochemical synthesis (MCS) and detonation-gas spraying. The method of mechatronic synthesis provided in a dry inert atmosphere the formation of the structure and phase composition of the powder mixture, which, as a result of selective interaction, due to thermodynamic and diffusion characteristics, consisted of micro volumes of several nanocomponents, which had a microcrystalline type of the base structure stabilized by nanoscale inclusions of high-temperature stable strengthening phases. Structurally free magnesium carbide (MgC_2) was added to the base particles obtained in this way and mixed until the mixture was evenly distributed, ready for detonation-gas spraying of coatings.

Coatings from the obtained composite powders were applied by the detonation-gas method to prepared samples of 30XGSNA steel. The increase in adhesion strength, as a criterion for operability, was carried out by pre-applying a sublayer of vitreous sodium $\text{NaO}_2(\text{SiO}_2)_2$ to the working surface of the samples. The adhesion strength was determined by the pin method, which for magnesium coatings was up to 98 MPa with a porosity of almost 0.5%, along with this, after grinding the initial roughness was Ra 0.32-0.63. Testing of the sprayed samples was carried out according to the end scheme under conditions of distributed contact at normal temperature in the continuous sliding mode with a constant load of 10.0 MPa. The influence of the environment, sliding speed, and load were provided taking into account the maximum approximation of the processes of physicochemical mechanics of friction to real conditions in the zone of frictional contact interaction, in addition, the research program provided for a comparative analysis of the friction parameters of the proposed coatings with similar values. Wear of coatings such as WK15 and coatings made of alloyed chromium.

Research into technology-structure and structure-property relationships was based on a complex of modern physicochemical methods of structural-phase analysis., which included consideration of the zones of the surface layers at the macro- and microscopic levels. In this case, the comprehensive research methodology included metallography (optical microscope "Neophot-32" with a prefix); particle size of the powder mixture (laser meter Analysette 22 Nano Tec plus; durometric analysis (hardness tester M-400 from LECO); scanning electron microscopy (scanning electron microscope JSM-840); X-ray structural phase analysis (diffractometer DRON-UM1).

Research results

Contact interaction during friction represents a complex sequence of mutually determined influences of both external and internal factors that determine the regularities of friction and wear processes and determine the degree and gradients of elastic-plastic deformation, temperature, activation level and a number of associated phenomena and are ultimately responsible for the leading type of wear.

The general results of coating tests (fig. 1) are presented in the form of graphs of functional averaged values of wear intensity and friction coefficients obtained in a field of monotonically increasing sliding velocities at constant the tensile load was 10.0 MPa.

Synthesis and research of ultra-strong ceramics based on ternary compounds, in particular magnesium boride, are being studied quite intensively, however, the possibilities of the latter and its complex of tribotechnical properties, despite efforts and individual achievements, have not been fully clarified in this regard to this day.

The analysis of the microstructure and elemental composition, carried out on the "Camebax SX" installation, shows that the synthesis products are heterogeneous and the main component is the chemical compound of aluminum, magnesium and boron (AlMgB_4). At the same time, a high background was found at small diffraction angles, which indicates the presence of an amorphous phase corresponding to boron and, possibly, highly boron-containing amorphous phases of aluminum and magnesium. In addition to the structural bases corresponding to the AlMgB_4 compound, reflexes of such phases as AlB_{12} , AlMgB_4 , AlB_2 were found, and $\text{Al}_{14}\text{Mg}_{13}$ imprints are present. It is important to note that the presence of a large number of phases with different complex crystal lattices makes it difficult to sufficiently determine the concentration of the target phase AlMgB_{14} . Quantitative phase separation is no less difficult, since the reflexes of the compounds present have close interplanar positions and are located at the same diffraction angles, as a result of which they overlap and complicate the determination of the real picture.

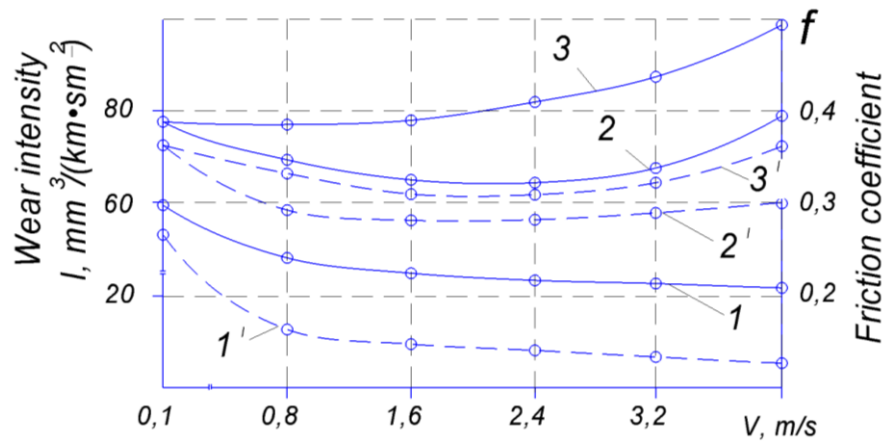


Fig. 1. Dependence of wear intensity (1, 2, 3) and friction coefficients (1', 2', 3') of coatings based on magnesium compounds (1, 1'), WK15 (2, 2'), alloyed nichrome (3, 3') at $P=10.0$ MPa.

It was found that the microstructure of the coating is finely dispersed and constitutes a heterogeneous mixture, with the dominant phase mainly being nanosized inclusions of silicon carbides (SiC) in the presence of titanium carbides (TiC), zirconium (ZrC) and an almost uniform amount of inclusions of zirconium borides (ZrB₂), chromium (CrB₂), magnesium (MgB₂) and a fine aggregate of strengthening compounds, which are stable silicide compounds of the Cr₂Si₃, Zr₃Si₂, TiSi, Mg₂Si type. Intermetallic formations were also identified, which contribute to the stabilization of the structure and have the form of spherical nanoparticles of the NiTi, NiAl, ZrCr₂ type. In addition, fragments of ternary compounds in the form of MgSiC, Ti₃SiC₂, ZrSiC were found, which also have high thermodynamic properties. As defined, AlMgB₁₄ is a complex compound that has the property of dissolving titanium and zirconium carbides, forming solid solutions with high hardness. A qualitative determination of the structure was implemented through the microhardness of individual areas. Thus, local zones with a microhardness of 21-26 GPa correspond, in our opinion, to AlMgB₁₄ compounds strengthened by carbide phases, while micro areas determined by a microhardness of 5.5-6.8 GPa are probably titanium carbo-silicides, and zones with a microhardness of about 12.0 GPa are titanium carbides, the value of 8.1 GPa most likely corresponds to titanium silicides, and the microhardness of 9.3-10.2 GPa is close to the values of magnesium oxides. Thus, the structural-phase formations of coatings based on ternary magnesium boride (AlMgB₁₄) include both chemical compounds, solid solutions, and mechanical mixtures, which are characterized by increased parameters of hardness, strength, wear resistance, significant temperature properties, and chemical inertness.

The formation of the structural-phase composition of coatings, as shown by the tests, is determined not only by the ratio of components, temperature, dispersion, but also depends on their defectivity and external conditions. It is certainly an axiom that tribochemical processes must occur when molecules in the conditions of interaction receive the necessary activation energy. Endothermic reactions do not occur at all without activation. The interaction of SiC with Mg, which is formed during the thermal decomposition of structurally free magnesium carbide and depends on the process temperature, is accompanied by the formation of magnesium silicide and magnesium acetylide, the latter under thermomechanical influence stimulates the formation of graphite through the intermediate dimagnesium trisilicide by the reaction type $2SiC + 5Mg \rightarrow 2Mg_2Si + MgC_2$, $MgC_2 \rightarrow Mg_2C_3 \rightarrow Mg + C$. We note that under thermodynamic influence, the presence of a catalyst in the structure in the form of Al, Ni affects the decomposition of magnesium carbide. In addition, the presence of nickel carbide (Ni₃C), which is thermodynamically unstable, causes an exothermic decomposition reaction into metallic nickel and carbon in the form of graphite: $Ni_3C \xrightarrow{t} 3Ni + C$. The decomposition temperature is not fixed and depends, in particular, on the particle size. For nanoparticles, due to their high surface energy, the decomposition onset temperature is 200-300 °C.

The basis of physical phenomena initiating the mechanism of decomposition of carbide graphite are structural transformations in the solid phase caused by thermal influences. Factors determining the quality level of thermomechanical carbide graphitization include the degree of dispersion of structural components, external pressure, operating temperature, including the temperature in the contact zone, the presence of elements initiating decomposition processes, as well as the influence of the environment (in a vacuum, the probability of the amount of graphite increases), in addition, internal factors associated with the composition of the material, its structure and the presence of defects.

The passivating functions of secondary structures are performed by the finely dispersed microstructure of the surface layer, which constitutes a multiphase mixture with the convincing presence of thermodynamic compounds in the form of simple and complex stable oxides such as MgO, Al₂O₃, SiO₂, Mg₂TiO₄ and others. Binary oxides in the form of MgO-TiO₂, MgO-ZrO₂ have also been identified, and the possibility of the presence of ternary compounds based on magnesium oxide, such as MgO-ZrO₂-TiO₂, MgO-Al₂O₃-TiO₂, is not excluded.

The solubility of silicon dioxide (SiO_2) in magnesium oxide (MgO) is very low, but they interact to form magnesium silicates Mg_2SiO_4 or MgSiO_3 within heterogeneous structures. The reaction formula in the presence of carbon is: $2\text{MgO}_2 + \text{SiC} + 2\text{C} \rightarrow 2\text{Mg} + \text{SiO}_2 + 3\text{C} - \text{Mg}_2\text{SiO}_4$.

Electron microscopic study of coatings under friction loading conditions revealed that an ultradisperse structure with a fragment size of the order of 25-40 nm is formed in a thin (~15 nm) surface layer.

Analysis of electron diffraction patterns from the friction surface proves that diffusion halos reflect an object with an ultra-disperse structure, and textured maxima indicate a directional orientation of the specified structure. The emergence of an ultra-disperse structure on the friction surface proves that the plastic deformation of the surface layer is carried out by a rotational mechanism, due to the relative sliding of fragments of ultra-disperse structures (fig. 2).

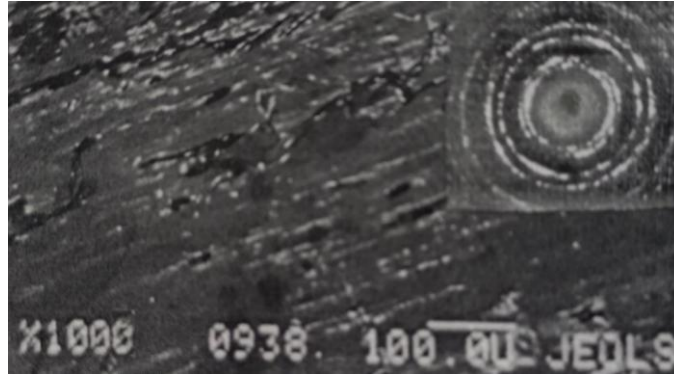


Fig. 2. Microscopic image of the structure of the local micro volume of the surface layer and micro electron diffraction pattern of a composite coating based on magnesium ternary boride (structure at a depth of 2-3 microns).

But, besides the ternary compound of aluminum, magnesium, boron (AlMgB_{14}) plays an important role in the surface complex of graphite-oxide structures, which complements the properties of the surface film, providing an additional increase in wear resistance while reducing friction coefficients. The results of X-ray structural analysis reveal the occurrence of activated phase transformations in magnesium structures, causing the formation of new phases, presumably due to intermediate compounds in the chains of transformations, the final product of which is, for example, oxides (Al_2O_3 , CrO_3), carbides (Al_4C_3 , Cr_7C_3), magnesium-based intermetallics. However, the ultradisperse structure determines their X-ray amorphousness. The presence of these phases indirectly confirms the high hardness.

When the sliding speed increases from 1.5 m/s, the specific work of wear reaches almost 104 kJ/mm^3 , which provides the necessary and sufficient conditions for the thermal decomposition of magnesium carbide and, as a result, fragments of structurally free graphite appear on the friction surface (fig. 3). The shape of the particles of the graphite structure is close to scaly, consisting of polydisperse crystallites oriented in the direction of friction. The basis of the physical phenomenon that constitutes the mechanism of thermal decomposition of carbide phases is the process of structural transformation in the solid phase. The main factors that determine the limiting values of thermodynamic graphitization processes are the level of dispersion of structural components, specific pressure, operating temperature, ambient environment, initiating elements (C, Si, Ni, Al), in addition, internal factors are determined by the composition, structure, presence of defects, etc.

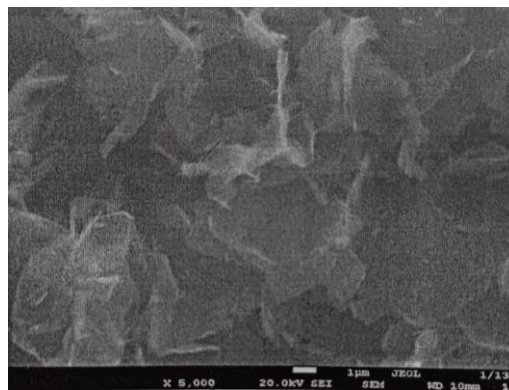


Fig. 3. Image of the local location of the structure of the α -graphite surface film ($\times 500$).

Self-lubrication of composite coatings of magnesium compounds depends on the formation of a graphite film (fig. 4). At test speeds of 2.8 m/s, the surface self-lubricating graphite-oxide film completely occupies the friction area and, at the same time, is a layer with the overwhelming majority of polydisperse graphite. At the same

time, the higher the temperature, the greater the amount of carbide graphite turns into a self-lubricating antifriction film and the longer the contact mating areas interact, the more graphite is formed.

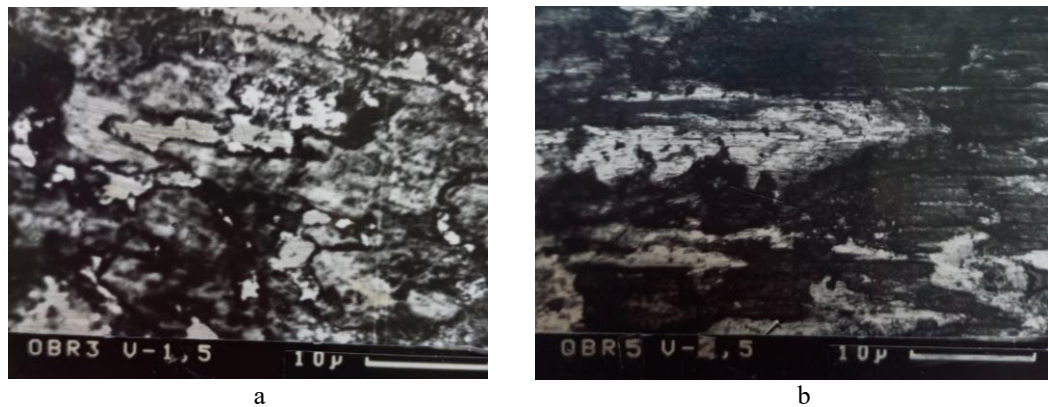


Fig. 4. Surface topography during the formation of a graphite film: a) $V = 0.18$ m/s; b) $V = 2.8$ m/s.

Thus, the studied coatings based on magnesium compounds form self-lubricating ordered dynamic structures during friction. The self-lubricating function is provided by the triple additive interaction of both magnesium carbide, which is the source of carbide graphite formation, and layered oxide structures, which are characterized by low shear resistance, and the presence of effective aluminum-magnesium-boron compounds. The established complex of self-lubricating surface structures under friction conditions has an effective ability to self-heal and self-regulate. The real effect ensures the minimization of friction and wear parameters.

From the point of view of structural thermodynamics, the systemic ordering of surface films that are self-formed due to transformations can be considered as adequate elementary physicochemical processes and adaptation mechanisms in the process of structural adaptability [18].

The problem of coating quality is associated with the assessment of reproducibility and optimization of the technological process of spraying. To obtain high-quality coatings by optimizing the technological process, processing of technological parameters was implemented, including the particle size distribution, loading depth, barrel filling degree, working gas ratio and spraying distance [19]. Thus, by controlling the technological process of forming coatings based on magnesium compounds, it was possible to implement not only the desired chemical composition, but also to obtain a predicted structure during spraying, which has an optimal set of properties that ensure the stability of structural adaptability. At the same time, the possibility of obtaining constant quality was achieved, namely, the variation of strength and plastic properties in samples of one batch was stably about 5-10%.

As can be seen from the test results, the control coatings are significantly inferior in tribotechnical properties compared to the developed self-lubricating coatings made of magnesium compounds.

Thus, the detonation coatings developed on the basis of magnesium compounds, capable of self-lubrication, are characterized by high antifriction properties and, in terms of operational potential, open up the possibility of using them in obtaining competitive systems for tribotechnical purposes. The set of results obtained [20, 21] allows us to recommend composite coatings from magnesium compounds to increase the service life of parts made of nickel alloys, including, for example, disks, working and nozzle blades, combustion chamber pipes, turbine disks, etc. Their use is justified for increasing the technical level and efficiency of repair work when restoring worn parts, which allowed us to significantly increase wear resistance and solve the problem of restoring previously unrepaired parts. The proposed coatings, as evidenced by the test results, ensure the operational reliability of tribotechnical connections in accordance with the conditions that are put forward for new competitive materials of antifriction coatings obtained by the detonation method. Therefore, composite coatings developed on the basis of ternary magnesium carbide can be considered as an alternative to other promising materials for operation in components of modern technology, including aerospace.

It should be noted that the developed composite powder based on magnesium reagents for forming antifriction self-lubricating coatings can be used for any technological methods using powder materials.

Finally, we will determine that the development and testing of magnesium coatings, despite economic difficulties, is a necessary component of the technical and social development of both science and society as a whole.

Conclusions

Composite coatings based on ternary compounds of aluminum, magnesium, boron, characterized by low and stable coefficients of friction and wear intensity, have been developed and investigated. In the test mode at a load of 10.0 MPa, the developed coatings have friction parameters significantly lower than those of control coatings by 3.5-8.0 times.

Based on the results of mechanochemical technology, the formation of the composite structure and phase composition of a powder mixture of magnesium compounds for detonation-gas spraying was synthesized.

The optimal mode of spraying of the magnesium composition has been worked out, which reproduces not only the planned chemical composition, but also provides a predicted structure, which modernizes the friction surface and ensures guaranteed quality of coatings. At the same time, it is emphasized that during spraying, the variation of the strength and plastic properties of coatings in samples of one batch is stable and amounts to 5-10%.

The structural and phase composition of coatings based on magnesium compounds has been established as a multicomponent fine-grained aggregate with an almost uniform distribution of finely dispersed strong inclusions of carbides, borides, silicides and intermetallic formations in the presence of double and ternary magnesium compounds, which are generally characterized by increased thermodynamic properties, high wear resistance, hardness, strength and low coefficient of friction with significant chemical inertness.

The developed self-lubricating composite coatings based on magnesium compounds extend the achievements of modern tribotechnical materials science. The studied compositions, which are capable of self-lubrication, can be used both for strengthening and for high-quality restoration of worn parts by any technological methods using powder materials.

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Щепетов В.В., Фіалко Н.М., Бись С.С. Тверді мастильні нанопокриття на основі сполук магнію

Представлено результати досліджень тертя та зносу детонаційних композитних нанопокриттів на основі потрійної сполуки алюміній-магній-бор в умовах випробувань з постійним навантаженням у полі швидкості ковзання. Їх структурно-фазовий склад та пасивуючий комплекс поверхневих оксидних структур визначено за допомогою сучасних методів фізичного аналізу. Встановлено, що параметри інтенсивності зносу та коефіцієнти тертя мінімізуються завдяки сукупності поверхневих структур, які регенерують стабільний самозмащувальний структурований шар в умовах тертя. Визначено фактори, що впливають на формування динамічної рівноваги самозмащувального шару, що має ефективну здатність до самовідновлення. Водночас, безперервний захисний шар екранує процеси молекулярно-адгезійної взаємодії та блокує розвиток неприйнятних явищ руйнування.

Ключові слова: поверхневі структури, карбідний графіт, знос, проміжний шар, фазовий склад, самозмащування.