Magnesium Reagents in the Synthesis of Magnesium Self-Lubricating Coatings

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Abstract: The results of studies of friction and wear of detonation composite coatings based on ternary magnesium boride under constant load over a range of sliding speeds are presented. Their structural and phase composition and the complex of surface oxide structures were determined using modern physical analysis methods. It has been established that the minimization of wear intensity and friction coefficients is due to the formation of a set of surface structures capable of regenerating a stable self-lubricating structured layer under friction conditions due to the ternary additive interaction of carbide graphite, complex oxide structures, and active aluminum- magnesium-boron compounds. Factors influencing the formation and maintenance of the dynamic level of the self-lubricating layer have been identified as a multiscale surface interaction phenomenon with an effective capacity for self-renewal and self-regulation. At the same time, the continuous protective layer of surface structures shields the adhesive-molecular interaction of contact combinations and blocks the development of unacceptable failure processes.

Keywords: Oxide structures, Carbide graphite, Wear, Interlayer, Phase composition, Self-lubrication, Physical analysis.

1. INTRODUCTION

The results of friction and wear studies for detonation composite coatings based on magnesium aluminum tetradecaboride under constant load testing across a range of sliding velocities are presented. Physical analysis methods were applied to their structural and phase composition and the complex of surface protective structures. It has been established that the minimization of wear rate and friction coefficients is driven by collective surface structures capable of regenerating a stable self-lubricating structured interlayer under friction conditions through the triple additive interaction between carbide graphite, complex oxide structures, and active aluminum-magnesium-boron compounds. The factors influencing the dynamic formation of a self-lubricating layer have been identified as a large-scale phenomenon of surface interaction. At the same time, the protector of surface structures shields the adhesive-molecular interaction and blocks the development of unacceptable deterioration processes.

Reliability and service life of industrial equipment are largely determined by the friction and wear phenomena. In contemporary tribological literature, research trends focusing on various types of reinforcing structures and their nanoscale characteristics have become widespread [1-3].

However, practical applications pose specific challenges that remain to be addressed. The most reliable measure for wear mitigation continues to be the use of diverse lubricants in friction units. Given the advancements in nanomaterials, there is a growing need to develop and characterize novel layered materials.

From this perspective, graphite has always been considered a classic representative of a class of objects whose planes exhibit weak adhesive properties and it accounts for its use as an antifriction agent.

In recent years, significant progress has been made in the application of solid lubricants. Solid lubricant-containing coating is one innovative and most promising antifriction materials, especially in self-lubricating conditions [4].

The development of antifriction self-lubricating nanostructured coatings aligns with current priorities in tribological materials science, aimed at improving the quality of moving joints and developing applied scientific solutions to enhance the efficiency of high-quality manufacturing technologies.

2. PURPOSE OF THE STUDY

Investigation of friction patterns and wear of magnesium-based coatings, study of their structural-phase composition, analysis of structure formation in antifriction surface layers, and consideration of their influence on self-lubrication processes.

3. MATERIALS AND RESEARCH METHODS

The primary ceramic magnesium compounds, proposed by the authors as powder reagents, are based on magnesium aluminum tetradekaboride

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(AlMgB14), which is characterized by high mechanical and thermal properties. To obtain a composite mixture, alloying impurities of silicon (Si), nickel (Ni), titanium (Ti), zirconium (Zr), and carbon (C) were added to the base material using a specific technology.

The choice of alloying elements was governed by tribological principles, enabling systematically to integrate the findings obtained into a unified framework of magnesium compounds with high tribotechnical properties. The subsequent production of high-quality self-lubricating coatings involved modern technologies, including mechanochemical synthesis (MCS) and detonation gas spraying. The MCS method, conducted in a dry inert atmosphere, ensured the formation of the powder mixture's structure and phase composition. Due to selective interaction determined by thermodynamic and diffusion characteristics, the mixture consisted of micro-volumes of several components featuring a microcrystalline base structure and nanoscale inclusions of high-strength stable reinforcing phases. Structurally free magnesium carbide (MgC_2) was added to the resulting base particles and mixed until a uniform distribution was achieved, making the mixture ready for detonation gas spraying. The resulting ceramic composite powders were deposited via the detonation gas method onto prepared 30KhGSNA steel specimens.

To improve adhesive strength as a performance criterion, a thin sublayer of vitreous sodium silicate $Na_2O{\cdot}(SiO_2)_2$ was pre-applied to the working surface.

Bond strength, determined by the pin method, reached up to 97 MPa with a porosity of nearly 0.5%, while the initial surface roughness after grinding was Ra 0.32–0.63 μm.

The sprayed samples were tested using a face-to-face scheme under distributed contact conditions at room temperature in continuous sliding mode under a constant load of 10.0 MPa.

The environmental influence, sliding velocity, and loads were modeled to closely approximate the physicochemical mechanics of friction to real-world conditions in the frictional contact zone. Furthermore, the research program included a comparative analysis of the friction parameters of the proposed coatings against analogous values for WK15-type coatings and alloyed chromium coatings.

The study of technology-structure and structure-property relationships was based on a complex of modern physicochemical methods of structural-phase analysis, involving the examination of surface layer zones at macro- and microscopic levels.

The integrated research methodology included metallography (Neophot-32 optical microscope with a special attachment); particle size measurement of powder mixtures (ANALYSETTE 22 Tec plus); durometric analysis (LECO M-400 hardness tester); scanning electron microscopy (JSM-840); and X-ray structural and phase analysis (DRON-UM1 diffractometer).

4. RESULTS AND DISCUSSION

Frictional contact interaction represents a complex sequence of mutually determined impacts from both external and internal factors. These factors govern the patterns of friction and wear, determining the degree and gradients of elastoplastic deformation, temperature, activation levels, and a range of associated phenomena, ultimately dictating the dominant wear mechanism.

The overall test results of the coatings (Figure **1**) are presented as functional averaged plots of wear rates and friction coefficients obtained within a field of

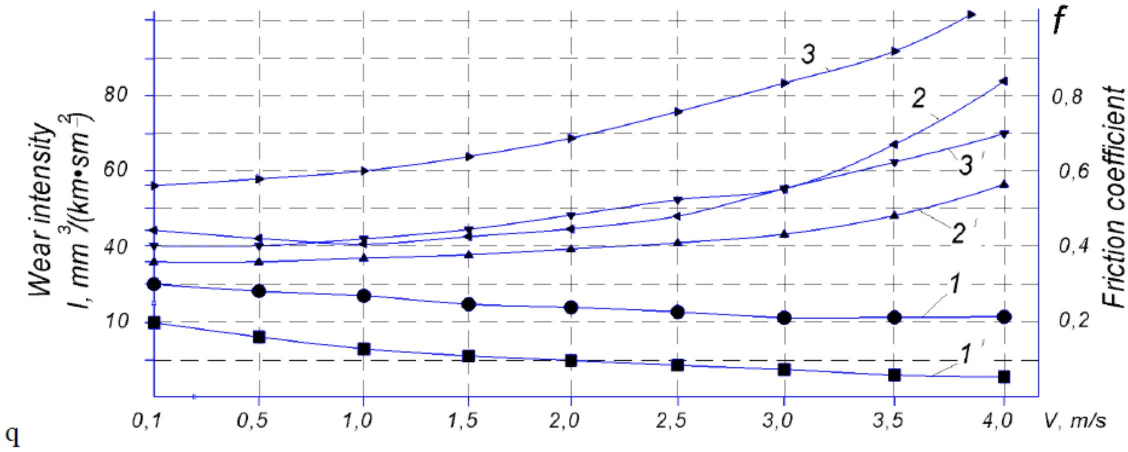


Figure 1: Dependence of wear intensity (1, 2, 3) and friction coefficients (1^{I}, 2^{I}, 3^{I}) of coatings based on magnesium compounds (1, 1^{I}), WK15 (2, 2^{I}), and alloyed nichrome (3, 3^{I}) at P = 10.0 MPa.

monotonically increasing sliding velocities under a constant load of 10.0 MPa.

The synthesis and investigation of superhard ceramics based on ternary compounds, specifically magnesium boride, are conducted quite intensively. However, despite significant efforts and individual achievements, the potential of the latter and its complex of tribotechnical properties have not yet been fully realized.

Microstructural and elemental composition analysis performed on a Camebax SX microanalizer indicates that the synthesis products are heterogeneous, with the primary component being the chemical compound of aluminum, magnesium, and boron ($AlMgB_{14}$). Additionally, a high background at small diffraction angles was detected, indicating the presence of an amorphous phase corresponding to boron and, potentially, high-boron amorphous phases of aluminum and magnesium.

In addition to the structural bases corresponding to the $AlMgB_{14}$ compound, reflections of phases such as AlB_{12}, $AlMgB_4$, and AlB_2 were identified, along with traces of $Al_{14}Mg_{13}$.

It is important to note that the presence of numerous phases with various complex crystal lattices complicates the accurate determination of the targ*et a*$lMgB_{14}$ phase concentration.

Quantitative phase separation is no less challenging, as the reflections of the present compounds have close interplanar spacing values and are located at identical diffraction angles; consequently, they overlap, obscuring the actual structural picture.

It has been established that the coating microstructure is fine-grained and consists of a heterogeneous mixture, which in composition is close to dispersion-strengthened composites. The dominant phase primarily consists of nanoscale inclusions of silicon carbides (SiC), alongside titanium carbides (TiC) and zirconium carbides (ZrC), with a nearly uniform distribution of titanium boride (TiB_2) and magnesium boride (MgB_2) inclusions. Notably, the presence of zirconium boride (ZrB) was not observed.

Simultaneously, a fine assembly of reinforcing compounds was found, representing stable silicides such as Zr_3, Si_2, TiSi, and Mg_2Si. Additionally, intermetallic formations that contribute to structural stabilization were identified as nanoparticles with a nearly spherical morphology, specifically NiTi and $ZrCr_2$.

Furthermore, fragments of ternary compounds such as MgSiC, Ti_3SiC_2, and ZrSiC were detected, which are also characterized by high thermodynamic properties. As determined, $AlMgB_{14}$ is a complex compound in which titanium and zirconium carbides act as reinforcement agents in the form of distinct, stable nanophases.

An attempt was made to qualitatively determine the structure by measuring the microhardness of local areas. Local zones with a microhardness of 23–27 GPa are believed to correspond to $AlMgB_{14}$ compounds reinforced with carbide phases. Meanwhile, micro-areas with microhardness levels of 5.5–6.8 GPa likely represent titanium carbosilicides, while zones with a microhardness of approximately 12.0 GPa correspond to titanium carbides. A value of 8.1 GPa most likely corresponds to titanium silicides, and a microhardness of 9.3–10.2 GPa is close to the values for magnesium oxides. Thus, the structural-phase formations of coatings based on the ternary magnesium boride ($AlMgB_{14}$) consist of both chemical compounds and solid solutions, and mechanical mixtures. These are characterized by enhanced hardness, strength, wear resistance, significant thermal properties, and chemical inertness.

As tests have shown, the formation of the structural-phase composition of coatings is determined not only by the ratio of components, temperature, and dispersion but also depends on their defects and external conditions.

It is an axiom that tribochemical processes occur when molecules acquire the necessary activation energy under interaction conditions.

An endothermic reaction, generally, does not occur without activation. The interaction of SiC with Mg, the latter being formed during the thermal decomposition of structurally free magnesium carbide and depending on the process temperature, is accompanied by the formation of magnesium silicide and magnesium acetylenide. The latter, due to thermomechanical influence, stimulates the formation of graphite via an intermediate dimagnesium tricarbide according to the reaction:

$$2SiC+5Mg\rightarrow 2Mg_2Si+MgC_2$$

$$MgC_2\rightarrow Mg_2C_3\rightarrow Mg+C$$

It should be noted that under thermodynamic influence, the presence of Ni as a catalyst in the structure affects the decomposition of magnesium carbide. Furthermore, the presence of thermodynamically unstable nickel carbide (Ni_3C) triggers an exothermic decomposition reaction into metallic nickel and carbon in the form of graphite. The decomposition temperature is not fixed and depends, in particular, on the particle size.

It has been established that, due to high surface energy, the onset temperature of decomposition for nanoparticles typically ranges between 200 and 300°C. The underlying physical phenomena triggering the decomposition mechanism of carbide graphite are solid-state structural transformations induced by thermal effects.

Factors determining the quality of thermomechanical carbide graphitization include the dispersion degree of structural components, external pressure, operating temperature, contact zone temperature, the presence of elements initiating decomposition, and environmental influences (*e.g.*, vacuum increases the graphite yield), as well as internal defect factors.

The passivating functions of secondary structures are performed by the fine-grained microstructure of the surface layer, which constitutes a multiphase mixture with a significant presence of thermodynamic compounds such as simple and complex stable oxides (MgO, SiO_2, Mg_2TiO_4, etc.). Binary oxides like $MgO{-}TiO_2$ and $MgO{-}ZrO_2$ were also identified, and the probability of ternary compounds based on magnesium oxide, such as $MgO{-}ZrO_2{-}TiO_2$ and $MgO{-}NiO{-}TiO_2$, is not excluded. The solubility of silicon dioxide (SiO_2) in magnesium oxide (MgO) is negligible, yet they interact to form magnesium silicates (Mg_2SiO_4 or $MgSiO_3$) within heterogeneous structures.

In the presence of carbon, the reaction formula is:

$$2MgO+SiC+2C \rightarrow 2Mg+SiO_2+3C \rightarrow Mg_2SiO_4$$

Electron microscopic examination of the coatings under frictional loading revealed that an ultradispersed structure with fragment sizes of approximately 25–40 nm forms 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 ultradispersed structure, while textured maxima indicate a directional orientation of said structure. The emergence of an ultradispersed structure on the friction surface demonstrates that plastic deformation of the surface layer occurs via a rotational mechanism due to the relative sliding of ultradispersed structural fragments (Figure **2**).

In addition to the surface complex of graphite-oxide structures, the ternary compound of aluminum, magnesium, and boron ($AlMgB_{14}$) plays a critical role, complementing the properties of the surface film and providing a further increase in wear resistance while reducing friction coefficients.

X-ray diffraction analysis of activated phase transformations in magnesium structures indicates the formation of new phases, presumably through intermediate compounds in transformation chains. The final products of these chains consist of, for example, oxides (Al_2O_3, CrO_3), carbides (ZrC, TiC), or base-metal intermetallics. However, due to their ultrafine structure, they exhibit X-ray amorphous characteristics. The presence of these phases is indirectly confirmed by high stiffness. As the sliding velocity increases from 1.5 m/s, the specific work of wear reaches nearly 104 kJ/mm^3, providing the necessary and sufficient conditions for the thermal decomposition of magnesium carbide. Consequently, fragments of structurally free graphite appear on the friction surface (Figure **3**).

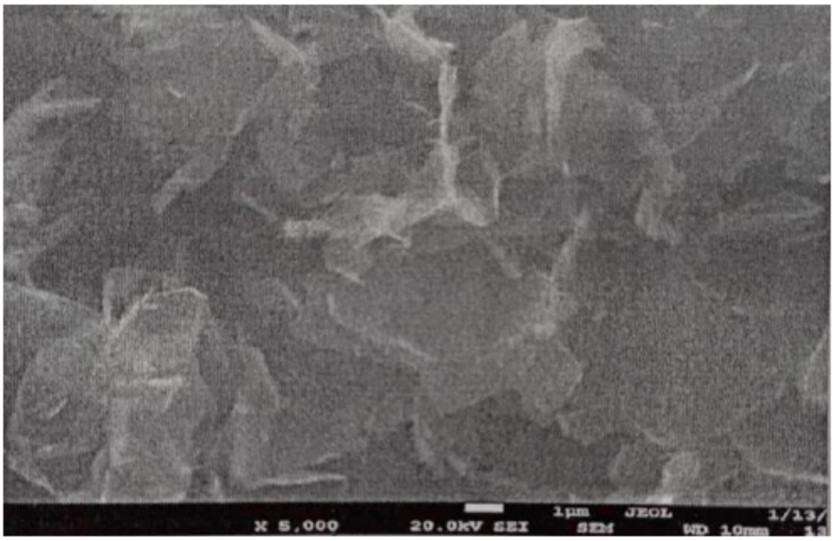
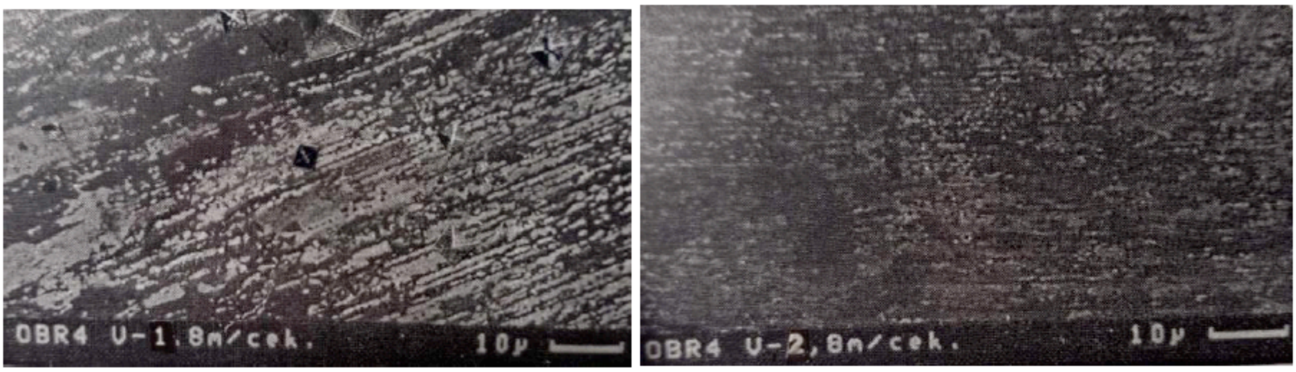


Figure 2: Image of a local area of the surface film structure of α-graphite (×500).



a

b

Figure 3: Surface topography during graphite film formation: **a**) V = 1,8 m/s; **b**) V = 2,8 m/s.

The shape of the graphite structure particles is close to flake-like, consisting of polydisperse crystallites oriented in the direction of friction. The physical phenomenon underlying the mechanism of thermal decomposition of carbide phases is a solid-phase structural transformation process. The primary factors determining the limit values of thermodynamic graphitization processes are the degree of dispersion of structural components, specific pressure, operating temperature, environment, and initiating elements (C, Si, Ni, Al); additionally, internal factors are driven by composition, structure, the presence of defects, etc. The self-lubricating capability of composite coatings derived from magnesium compounds depends on the formation of a graphite film (Figure **4**). At test velocities of 1.8 m/s, a self-lubricating graphite-oxide surface film completely occupies the friction area; this layer consists predominantly of polydisperse graphite. Simultaneously, higher temperatures accelerate the transformation of carbide-derived graphite into a self-lubricating anti-friction film. Furthermore, prolonged interaction between contact interfaces leads to increased graphite formation.

Thus, the investigated magnesium-based coatings form ordered self-lubricating dynamic structures during friction. This self-lubricating function is provided by a triple additive interaction between: magnesium carbide, which serves as the source for carbide-derived graphite; layered magnesium oxide structures, characterized by low shear resistance; the aluminum-magnesium-boron compound, which possesses an inherently low friction coefficient.

The regenerated complex of self-lubricating surface structures exhibits an effective capacity for self-recovery and self-regulation under frictional conditions. This mechanism ensures the stable minimization of friction and wear parameters.

From the perspective of structural thermodynamics, the systemic ordering of self-forming surface films via transformations can be viewed as fundamental physicochemical processes and adaptation mechanisms within the framework of structural compatibility [5].

The quality of the coatings is directly linked to the reproducibility and optimization of the thermal spray process. To obtain high-quality coatings via optimization of the manufacturing process, processing of technological parameters was implemented, including particle size distribution, loading depth, barrel fill level, working gas ratio, and spraying distance [6].

Thus, by controlling the optimal technological process for forming coatings based on magnesium-ceramic compounds, it was possible to reproduce not only the intended chemical composition but also to obtain a predictable structure with an optimal set of properties, ensuring the stability of the structural adaptability phenomenon. Furthermore, consistent quality was attained, with a data scatter of 5–10% in strength and ductility across the batch. Comparative test results indicate that reference coatings are significantly inferior in tribological performance compared to the developed self-lubricating magnesium-based ceramic coatings. Consequently, the magnesium-based detonation coatings developed in this study, which exhibit a self-lubricating effect, are characterized by high anti-friction properties. Their operational capabilities open new avenues for utilizing competitive tribological systems.

The aggregate results [7, 8] allow us to recommend magnesium-based composite coatings for extending the service life of nickel-alloy components, including discs, rotor and nozzle blades, combustion chamber liners, turbine discs, and similar parts.

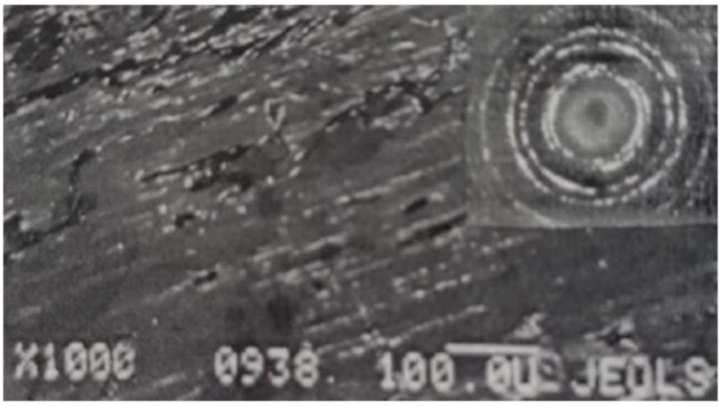


Figure 4: Microscopic image of the structure of the local microvolume of the surface layer and microelectronogram of a composite coating based on ternary magnesium boride (structure at a depth of 2-3 μm).

Their application is justified for improving technical standards and efficiency during the repair and restoration of worn surfaces, thus significantly increasing wear resistance and solving the problem of reclaiming components previously considered non-repairable.

As evidenced by test results, the proposed coatings ensure the operational reliability of tribological joints in accordance with the standards required for new competitive materials produced by detonation spraying. Therefore, composite coatings based on ternary magnesium carbide can be considered a viable alternative to other advanced materials for application in modern engineering assemblies, including aerospace applications.

It should be noted that the developed powder based on magnesium reagents for forming anti-friction self-lubricating coatings can be utilized in any technological method that employs powder materials.

Recently, the development and testing of magnesium coatings, despite economic challenges, has become an essential component of technical and social advancement for both science and society as a whole.

5. CONCLUSIONS

1. Composite coatings based on ternary compounds of aluminum, magnesium, and boron were developed and investigated. These coatings are characterized by low and stable friction coefficients and wear rates. Under test conditions with a load of 10.0 MPa, the developed coatings exhibit friction parameters 3.5 to 8.0 times lower than those of the reference coatings.

2. Employing mechanochemical technology, a composite structure and phase composition were synthesized in a magnesium-compound powder mixture intended for detonation-gas spraying.

3. The optimal spraying parameters for the magnesium composition were established, reproducing not only the target chemical composition but also ensuring a predictable structure that modifies the friction surface and guarantees coating quality. It is emphasized that the variation in the strength and plastic properties of the coatings within a single batch remained stable at 5–10%.

4. The structural-phase composition of the magnesium-based coatings was identified as a multi-component, fine-grained matrix with a nearly uniform distribution of finely dispersed, high-strength inclusions (carbides, borides, silicides, and intermetallic formations). The presence of binary and ternary magnesium compounds results in enhanced thermodynamic properties, high wear resistance, hardness, strength, and low friction coefficients combined with significant chemical inertness.

5. The developed self-lubricating composite coatings based on magnesium compounds advance the state of the art in modern tribological materials science. These self-lubricating compositions can be applied both for surface strengthening and high-quality restoration of worn parts using any technological methods that utilize powder materials.

CONFLICTS OF INTEREST

The author declared no conflicts of interest.

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