



## Self-lubricating glass composite magnesium carbide nanocoating

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### Abstract

The results of the study of the friction and wear characteristics of the developed nanostructured glass composite self-lubricating coatings are presented, the structural components which through magnesium carbide have a qualitative effect on the graphitization process due to the formation of a surface layer of carbide  $\alpha$ -graphite, which, when combined with surface oxides characterized by low shear resistance, performs the role of solid lubricants under friction conditions. The positive role of the glass phase in the form of aluminoborosilicate, which affects the tribotechnical properties of coatings, has been established. It has been determined that the increase in adhesion strength is achieved by forming a surface layer of vitreous sodium silicate during spraying. It has been found that the intercalation of the graphite layer with dispersed particles of the surface structure does not significantly affect the tribotechnical characteristics. The developed nanostructured glass composite coatings showed high antifriction characteristics.

**Keywords:** wear intensity, glass composite, nanostructure, wear, glass phase.

### Introduction

Preservation of operational characteristics limited by friction and wear, both of individual components and of technical systems as a whole, can be ensured by modern surface engineering tools that implement the basic principle of minimum costs for maximum results. Structural engineering methods that use modification through the use of solid lubricants have achieved significant success in recent years in ensuring antifriction properties of contact joints. Coatings containing solid lubricants are among the innovative and most promising antifriction materials, the high quality of which is especially noticeable in conditions operation where traditional liquid lubricants are not very effective [1,2]. They are used in various fields of engineering from lubricating precision aircraft mechanisms to preventing seizure of threaded joints [3,4].

Development of anti-friction nanostructured glass composite coatings, having self-lubricating properties due to solid lubricating compounds based on graphite, meets modern priorities of tribotechnical materials science, aimed at increasing the wear resistance of moving joints and development scientific and applied solutions in the interests of improving the efficiency of the application of high-quality production technologies [5,6].

### The purpose of the work

Research on the structural-phase composition and surface secondary structures of glass composite nanocoatings and their effect on the self-lubrication process under friction conditions.

### Materials and research methods

As is known from the comparative characteristics of gas-thermal coatings, similar in structural and phase composition, the maximum operational properties are possessed by detonation-gas coatings [7]. Therefore, for spraying the studied coatings, the technology of detonation-gas spraying of nanostructured powders of the SiC-Ni-Cu-Al-Si-C composition with a uniform distribution of the aluminoborosilicate glass phase ( $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-B}_2\text{O}_3$ ) was used. Structurally free magnesium carbide ( $\text{MgC}_2$ ) was added to the nanoglass composition obtained by the mechanical method, after which it was mixed, ensuring its uniform distribution in the resulting powder mixture



ready for spraying. All components of the powder materials used in the work were taken from the mineral raw material base of Ukraine.

The self-lubricating properties of the coatings were evaluated when rubbing ring samples according to the end scheme under conditions of distributed contact in a continuous sliding mode at a constant load of 10.0 MPa. The influence of the environment, speed, and load implemented during the tests were selected taking into account the maximum approximation of the processes of physicochemical mechanics of friction to real conditions of contact interaction. In addition, the research program of nanostructured glass composite coatings with magnesium carbide provided for a comparative analysis of their parameters with similar values obtained during tests of coatings of the WK15 type and coatings sprayed with doped nichrome powder.

The study of contact joints, in which the processes of activation occur during friction, which determine the intensity of surface heterophase configuration reactions and tribochemical phenomena, was carried out by modern methods of physical analysis, which include metallography (optical microscope type Neophot-32 with a prefix), X-ray electron micro analyzer type "Camsan 4DW" with a program for the distribution of chemical elements. Determination of the phase composition of surface layers was carried out on a general-purpose X-ray diffractometer type DRON-3 with monochromatized  $\text{CuK}\alpha$  radiation.

The increase in adhesion strength, as a criterion for the performance of glass composite coatings, was carried out by preliminary application of a sub layer of vitreous sodium silicate  $\text{Na}_2\text{O}(\text{SiO}_2)_2$  to the working surface. The exclusion of unproductive losses and compliance with the measurement technology [8] determined the correctness of the obtained adhesion strength results, which amounted to 140-145 MPa.

### Research results and their discussion

The contact interaction of surfaces is a complex sequence of mutual influence of both external factors and internal transformations, the qualitative coordination of which reflects the commonality of quantitative patterns and determines their ordered causal relationship. According to the results of the interaction of coatings under friction loading, fig. 1 gives experimental values that reflect the averaged functional dependences of the wear intensity and friction coefficients, which change in time and stabilize after running-in, in the field of sliding speeds at a constant load equal to 10.0 MPa. As can be seen from the graph, in the entire range of tests at a monotonically increasing sliding speed, the minimum parameters of wear intensities and the corresponding friction coefficients are characteristic of glass-composite nano coatings (curves 1 and 1'). The structure of the glass composition, which determines their properties, consists of a finely dispersed mixture, which represents solid solutions and, mainly, intermetallic compounds in the presence of a glass phase. The constancy of the chemical composition and the constancy of the properties of technological spraying determine the stability of the structure of the coatings, the relative density of which reaches 98%. The cross-section of the glass composite nanocoating is presented in fig. 2. Metallographic analysis has established that the sprayed layer has a quasi-ordered lamellar appearance and fits tightly to the base material, completely copying the surface relief, while the accumulation of component oxides, as well as slag contamination, are virtually absent, and defects in the form of pores and cracks are not detected.

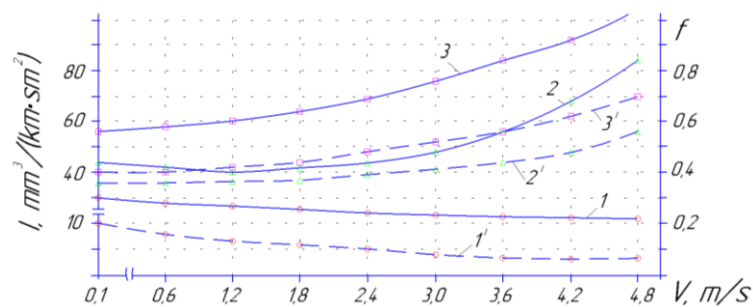


Fig. 1. Dependence of wear intensity (1, 2, 3) and friction coefficient (1', 2', 3') on the sliding speed of SiC-Ni-Cu-Al-Si-C+glass phase+MgC2 coatings (1,1'), WC-Co (2,2') and Ni-Cr-Al-B (3,3') on the sliding speed ( $P=10.0\text{MPa}$ ).

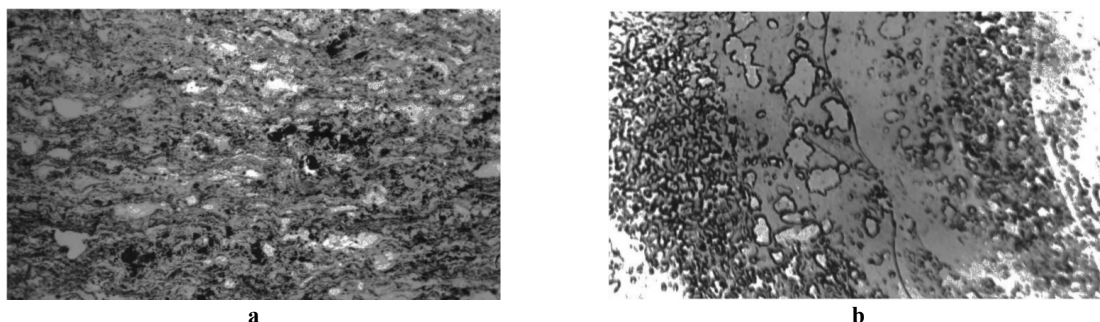


Fig. 2. Coating structure: a x 320; b x 5000

The micro geometry of the working surface of the nanocoatings under study, in combination with their physical and mechanical properties, determines their operational state. Studies have shown that during the running-in process, the initial technological relief disappears, the chemical composition, structure of the surface layer and its geometry radically change. It is possible to determine that running-in is one of the manifestations of the self-organization process, in which the quasi-relaxation of the surface structure from the equilibrium state will pass to a stable state. At the same time, a new surface quality is formed, characterized by the formation of balanced roughness, which is not only optimal for specific friction conditions, but also ensures stable wear in the entire range of tests.

The problem of coating performance is related to the quality of reproducibility and optimization of the spraying process. Changing the technological modes leads to changes in the properties of the coatings. To obtain high-quality coatings by optimizing the technological process, processing of technological parameters, such as particle size distribution, loading depth, barrel filling degree, working gas ratio, spraying distance [9], is implemented.

Synthesis and research of solid solutions based on refractory compounds, in particular silicon carbide, are being carried out quite intensively, however, the capabilities of the latter and its complex of tribotechnical properties are far from the expected results [10]. Thus, by controlling the technological process of forming glass composite nanocoatings, it was possible to implement not only the desired chemical composition, but also to obtain the predicted stable structure. Which optimizes the complex of properties that determine the manifestation 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 5-10%.

The developed glass composite is an antifriction material with a finely dispersed structure. It is generally accepted that elastic-plastic deformation is the main factor determining the development of the external friction process, and in addition, in our opinion, the formation of a gradient structure is a derivative of it. It can be said that the evolution of the structure during contact interactions has pronounced scale levels, and the processes occurring at different scale levels are mutually dependent. The layer-by-layer picture of plastic deformation, obtained by the diffraction method, reveals the main patterns of the formation of a scale structure and allows us to establish uniform transitions from a dispersed polycrystalline fragmented structure on the surface through intermediate textured layers to the initial crystalline structure inherent in the deep material. In the studied coating, as the friction surface approaches and the contact pressure and deformation intensity increase, the structure gradually changes to an ultra disperse one. At the same time, high contact compressive and shear stresses create conditions for the realization of significant plastic deformations in the surface layer of the coating material, which lead to the formation of ultra disperse structures.

This gives grounds to distinguish in the structure subjected to tribotechnical loads, a near-surface zone in which deformation processes developing in micro volumes in homogeneously form a specific layer at the near-surface level, in which structural-thermal activation causes a complex of physicochemical interactions that determine the accompanying and dominant type of wear. The near-surface zone has a structurally inhomogeneous fine-dispersed composition.

As shown by the results of micro-X-ray spectral analysis (MRSA) performed on the CamebaxSX installation, the basis of the nanoglass composition is silicon carbide of non-stoichiometric composition, along the grain boundaries of which silicate compounds are located, among which inclusions corresponding to the composition of silicon dioxide predominate, also in the carbide structure the role of dispersedly strengthened components is performed by  $\text{Al}_2\text{O}_3$  oxides distributed along the boundaries and intermetallic inclusions in the form of spherical nanograins. But the high thermo mechanical properties of SiC carbide are discredited by significant fragility. We have noted that the solid substitution solution formed by Al and SiC causes a slight distortion of the crystal lattice of the carbide, since the differences in the masses of Al and Si atoms are extremely small, as a result of which the micro hardness does not change, and the plasticity increases. Cu and Ni have a similar effect on the composition of SiC, which form solid substitution solutions by replacing Si atoms. The formation of phases in the coating, 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 undeniable that tribochemical interaction occurs when molecules receive the necessary activation energy. Endothermic reactions do not occur without activation at all. The interaction of SiC with Mg, which is formed during the thermal decomposition of structurally free magnesium carbide under running-in conditions and depends on the process temperature, is accompanied by the formation of magnesium silicide and magnesium acetylide, the latter under the influence of thermo mechanical interactions promotes the formation of graphite through the intermediate dimagnesium tricarbide ( $2\text{SiC} + 5\text{Mg} \rightarrow 2\text{Mg}_2\text{Si} + \text{MgC}_2$ ,  $\text{MgC}_2 \rightarrow \text{Mg}_2\text{C}_3 \rightarrow \text{Mg} + \text{C}$ ). It should be noted that under thermodynamic action, the presence of a catalyst in the form of Al in the structure promotes the decomposition of magnesium carbide. The basis of physical phenomena that initiate the mechanism of decomposition of carbide graphite are structural transformations in the solid phase caused by thermal influences. The factors that determine the quality level of thermo mechanical carbide graphitization, in our opinion, include the degree of dispersion of structural components, specific pressure, operating temperature, and temperature in the contact zone, the presence of elements that initiate 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 related to the composition of the

material, its structure, the presence of defects, etc. In fig. 3 the topography of the friction surfaces obtained at sliding speeds of 0.5 m/s and 0.17 m/s is given. The antifriction layer of graphite, as can be seen, covers almost the entire working surface with all its capacity, ensuring an increase in the actual contact area, contributing to a decrease in the specific load due to an increase in the support length due to filling and leveling micro-irregularities and fixing graphite micro particles in micro-cavities. The contact zone, which is the surface layer (initial scale level), which separates the coating material from the antifriction film consisting of polydisperse graphite particles, represents a deformed zone, which, according to the results of micro-X-ray spectral analysis performed on MAP-3 (probe diameter 1  $\mu\text{m}$ ), represents finely dispersed heterogeneous structural-phase compounds of the components. Among which the presence of Ni as a structural component is due to its specific properties, namely, at the spots of actual contact when the temperature reaches 450-500°C depending on the dispersion and external influences in the conditions of a local high-temperature field, Ni interacts with SiC, forming metal-enriched nickel silicides ( $\text{Ni}_2\text{Si}$ ). As a result, carbon is reduced, which is transformed into a solid phase of elementary polydisperse graphite colonies, combined into surface structures.

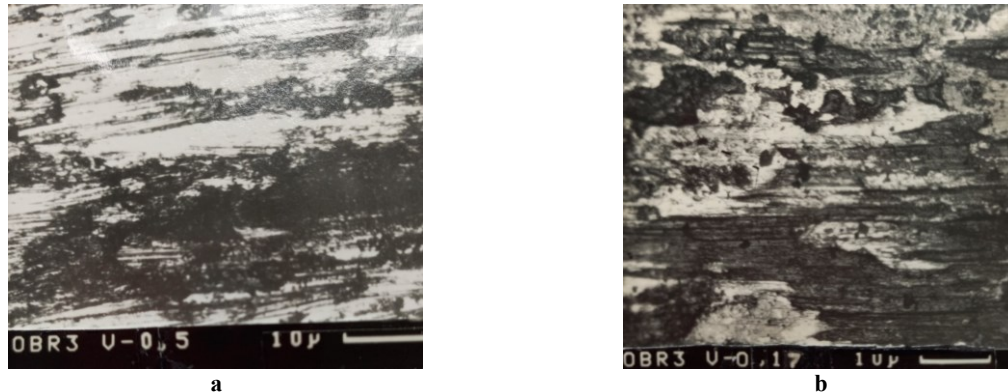


Fig. 3. Friction surface topography at formation of graphite film: a –  $V=0.5$  m/s; b –  $V=1.7$  m/s

But the dominant component of the formation of the antifriction surface layer, consisting of a carbon product - graphite, remains magnesium carbide. The value of the specific work of wear, characteristic of the initial moment of running-in, as calculations have shown, is up to 10  $\text{kJ/mm}^3$ , which is both a necessary and sufficient condition for the initiation of thermal decomposition of  $\text{MgC}_2$ , which leads to the formation of carbon in the form of a solid phase.

Using the natural ability of chemical elements to graphitize through the formation of carbide graphite, we obtained a high-quality, anti-friction layer that determines the operational properties of the coatings.

In the structural-phase study of glass composite coatings, the presence of intermetallic compounds based on Al and Ni of the  $\text{NiAl}$  and  $\text{Ni}_2\text{Al}_3$  type was noted, while monoaluminide, which is a high-temperature phase, has significant hardness, as measurements showed, most likely close to 3.8 GPa. The presence of an ordered solid solution based on nickel monoaluminide was also established, which has an Al content of ~ 20-25 wt. %, which causes increased plasticity. According to the results of elemental and X-ray phase analyses, the presence of a solid solution of Ni in Cu was noted, but their compounds were not detected. Solid solutions of Ni in Si and Si in Ni, as well as their intermetallics  $\text{Ni}_2\text{Si}$ ,  $\text{Ni}_3\text{Si}_2$ ,  $\text{NiSi}_2$  were established. In addition, the presence of small quantities of colonies of solid solutions of Si in Cu has been proven, and the formation of their chemical compounds such as copper silicates is also likely, since the structural micro hardness is significantly increased, but identifying them presented significant difficulties.

Aluminoborosilicate glass powders, the dispersion of which was 25-30 microns, in the process of mechanochemical treatment and thermo mechanical impact, as products of inorganic synthesis, caused, as was determined, along with the preservation of the initial components, the formation of new stable compounds from solid solutions of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  with an orthorhombic syngony, which is close to the structure of sillimanite, most likely this is lower mullite, obtained by a reaction as a result of the interaction of oxidation products of the initial components. From the point of view of glass-ceramic technologies, the greatest interest, in our opinion, is the presence of components that form refractory metal oxides and primarily Al and Si oxides. The presence of  $\text{B}_2\text{O}_3$ , which, as a result of partial oxidation, became part of the solid solution  $\text{Al}_2\text{O}_3\text{-B}_2\text{O}_3$ , was also established.

From the perspective of condensed matter physics, the addition of a glassy component affects the quality of the coating material through the structural state and, as practice shows, interest in these technical products is steadily growing. When studying glass compositions, their optimal composition was established experimentally, in which the rational use of glass structures contributes to increased heat resistance and chemical stability, in addition, the manifestation of high cohesive strength, increased density of nanocomposites, crack resistance and, with significant corrosion resistance, ensures high adhesion (more than 127 MPa) with materials of different chemical nature, in addition, the formation of a silicate barrier layer prevents mutual diffusion of structure-forming particles of the coating and the substrate.

The surface zone, directly adjacent to the friction surface and separating the coating material from the antifriction layer, consisting of polydisperse graphite particles, is the thinnest film with a thickness of several micrometers. Studies have shown that the pressure in it is uneven, and the areas of tensile and compressive stresses, which are inevitable under conditions of deformation of heterogeneous phases, are close in structure to a conglomerate of finely dispersed (quasi-amorphous) structures, having dimensions in the range of 5-15 nm, and are mechanical mixtures, without oxygen and oxide compounds of the structure-forming components. The effect of plastic deformation is associated not only with the dispersion of the surface zone, but also with the accumulation of defects that change its physicochemical properties, including reactivity, and affect the intensity of chemical reactions in the solid phase.

At the same time, the thermal conductivity of the finely dispersed conglomerate has increased porosity and forms a lower near-surface zone than that of the solid material, therefore the heating temperature of the finely dispersed fragments of the zone is higher than the temperature of the surface areas.

The temperature factor stimulates physicochemical processes, in particular, the reactive diffusion of structure-forming particles at the atomic-molecular level, which promotes the penetration of kinetically active components of the dispersed zone due to the weakening of the bond between polyarene planes into the interlayer space of graphite and, thus, the formation of intercalated graphite.

By the method of X-ray phase analysis, it was established that the intercalating elements in the subsurface zone-graphite system, at the initial stage of the process, appeared ions  $Mg^{2+}$ ,  $Al^{3+}$ ,  $Cu^{2+}$ , which randomly penetrate into the interlayer space of the graphite matrix. At sliding speeds of more than 3.0 m/s, intercalates of binary molecular compounds of these elements with oxygen were found in the layered graphite system. Their intercalation is accompanied by a sequence of repeating stages, which, when changing the tribotechnical parameters, are reversible and are characterized by a specific transformation of the structure and, first of all, an increase in the distance between the layers due to the influence of various types of interlayer defects and the penetration of intercalants. It should be noted that today there is no general model of intercalation that explains the electrochemical mechanism of the synthesis of layered systems. From an energetic point of view, the intercalation process, which represents reversible topotaxis chemical reactions, can be considered as an adequate mechanism for the self-organization of surface layers in the process of structural adaptability of the friction system.

We have established that the level of quantitative changes during the intercalation of the graphite layer, which determines the high level of antifriction, does not affect the qualitative values of tribotechnical parameters during the testing process to the expected extent.

Antifriction nanostructure glass-ceramic self-lubricating coatings have been developed, which contain magnesium carbide and structural components that promote surface graphitization, do not contain expensive and scarce components, meet environmental safety requirements, and provide high performance properties. From our considerations, the developed and researched self-lubricating glass composite nanocoatings can be considered as an alternative to other promising materials for operation in modern designs of tribotechnical systems. Their application most effectively for increasing the operational reliability of friction units, for example, moving parts of control mechanisms, cams, pairs with reciprocating movement, sliding supports, lever parts, high-speed and thermally loaded couplings, in which the use of traditional lubricants is undesirable.

The development of nanostructured glass-ceramic antifriction self-lubricating coatings, the justification of their structural components, the results of applied tests, and the ability to work in production conditions allows us to significantly expand the arsenal of achievements in modern tribotechnical materials science.

It should be noted that a nanostructured glass composite powder has been developed, which contains magnesium carbide, can be used to strengthen and restore worn parts of any shape and design, using any technological methods that use powder materials.

## Conclusions

1. The developed and investigated glass composite nanocoatings, which are characterized by stable and minimal values of friction coefficients and wear intensities under test conditions at a sliding speed of up to 5.0 m/s and a load of 12.5 MPa, have friction parameters significantly lower than those for control coatings by almost 3.5-8.0 times.

2. Through theoretical and experimental studies, the optimal structural-phase composition of nanostructured coatings of the SiC-Ni-Cu-Al-Si-C system, containing a glass phase of the  $SiO_2-Al_2O_3-B_2O_3$  type and structurally free magnesium carbide, was implemented. At the same time, to improve the adhesion strength, a sublayer of vitreous sodium silicate was applied to the substrate. At the same time, the role of the glass phase in the formation of glass compositions was studied, which contributes to an increase in the cohesive component, continuity and strength of the nanostructure, and increased chemical resistance.

3. It is noted that the assessment of the quality of the studied coatings is associated with the problem of reproducibility of the technological process. By controlling the spraying of glass composite powders, it turned out to be possible to provide not only the required chemical composition, but also to obtain a given nanostructure that optimizes the complex of properties that contribute to the stable manifestation of the minimization of tribotechnical

parameters in conditions of structural adaptability. It is also noted that the variation of strength and plastic properties in the sprayed samples was stable and amounted to 5-10%.

4. The physical mechanism and main factors determining the level of thermo mechanical graphitization are considered. The nature and regularities that determine the tendency of coatings to passivation are studied. It is noted that its implementation is carried out both due to solid-phase tribochemical reactions, which cause the formation of quasi-spherical polydisperse surface films integrated on the basis of carbide graphite and dispersed oxide compounds.

5. The structural and phase composition of glass composite nanocoatings was investigated by means of physicochemical analysis, their fine-dispersed structure was established, while it was emphasized that the components of the composite particles are solid solutions based on binary oxides and inclusions of chemical compounds of simple and complex carbides and intermetallics, as well as mechanical mixtures of component compounds. These components have increased temperature stability, significant hardness and strength, and chemical inertness.

6. Fundamental ideas about the formation and structure of antifriction surface structures based on dispersed carbide graphite have been supplemented, which has allowed us to expand the arsenal of achievements in modern tribotechnical materials science.

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**Щепетов В.В. , Фіалко Н.М. , Бись С.С.** Самозмашувальні склокомпозиційні нанопокриття з карбідом магнію.

Наведено результати дослідження характеристик тертя та зношування розроблених наноструктурних склокомпозиційних самозмашувальних покриттів, структурні складові яких через карбід магнію мають якісний вплив на процес графітизації за рахунок утворення поверхневого шару карбідного  $\alpha$ -графіту, який при спільній дії з поверхневими оксидами, що визначаються низьким зсувним опором, виконують в умовах тертя роль твердих мастил. Встановлено позитивну роль склофази у вигляді алюмоборосилікату, що впливає на триботехнічні властивості покриттів. Визначено, що підвищення адгезійної міцності досягається за рахунок формування при напилюванні поверхневого шару зі склоподібного силікату натрію. З'ясовано, що інтеркаляція графітового шару дисперсними частинками поверхневої структури суттєво не впливає на триботехнічні характеристики. Розроблені наноструктурні склокомпозиційні покриття показали високі антифрикційні характеристики.

**Ключові слова:** інтенсивність зношування, склокомполит, наноструктура, знос, скло фаза.