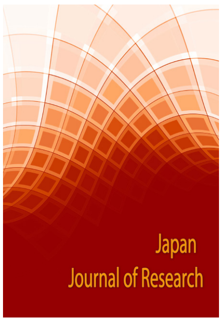
Japan Journal of Research

Original Article



Self-Lubricating Tribotechnical Nanocoatings Based on Magnesium Compounds

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- Received Date: 19 June 2026
- Accepted Date: 30 June 2026
- Publication Date: 02 July 2026

Keywords

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

Abstract

The paper scientifically substantiates and implements an integrated approach to creating antifriction self-lubricating composite nanocoatings based on magnesium compound, forsterite and magnesium-trialuminum carbide, deposited by detonation spraying. The results of friction and wear processes under various sliding velocities and a constant load of 12 MPa are presented. The structural-phase composition and passivating complexes on the friction surface were analyzed using modern physical methods. The results show that the minimization of wear intensity and friction coefficients is driven by the formation of a self-regulating structured layer. This layer maintains dynamic equilibrium due to the additive interaction of graphite (released from carbides), complex oxide structures, and passivating structures of the matrix phase.

It was established that the formation of a fine-grained surface structure (grain size of 20–35 nm) activates a rotational deformation mechanism, resulting in high hardness (12.0–15.0 GPa) alongside with low shear resistance. The magnesium-containing nanocoating exhibits a wear resistance 3.3 to 8.5 times higher than traditional tungsten-containing and nichrome coatings, thereby making them highly suitable for extreme operating conditions in the aerospace industry and mechanical engineering.

Introduction

Friction and its associated wear are among the oldest problems in technical systems, which remain highly relevant today. Although tribological research results are quite significant, they still fail to fully meet practical demands for creating durable and reliable friction units. A traditional technique to reduce contact surface wear involves creating various lubricating films that separate the rubbing surfaces [1]. Composite materials based on a metal matrix containing functional fillers for various purposes have become widespread [2]. To improve the antifriction properties of tribological composites, graphite, molybdenum disulfide, boron nitride, and other substances are commonly used. Metal-matrix composites incorporating polymer fillers, such as DU, DP, and DX materials, also retain their popularity [3].

However, developing new solutions that present equal technical and economic advantages for coatings remains critically urgent. The designed compositions operating without additional external lubrication must realize internal self-lubrication conditions during service, which reduce wear intensity to minimum values, completely eliminate the need for regular maintenance, and lower repair costs [4].

The development of new antifriction composite coatings that ensure the stable operation of machine friction units and mechanisms through the formation of surface structures, possessing a stable manifestation of minimized friction and wear parameters via self-regulation of structured surface layers with reactive self-lubricating properties, is one of the main tasks of tribological materials science and addresses urgent problems in modern manufacturing.

Purpose of the Work

The study of the structural-phase composition of a composite coating based on magnesium compounds, the investigation of its wear patterns, and the analysis of friction and self-regulation processes in structured antifriction surface structures possessing a stable self-lubricating effect.

Materials and Research Methods

When creating the powder composition, the authors utilized the synergistic properties of a mechanical mixture of forsterite (Mg_2SiO_4) with triple aluminum-magnesium carbide (Al_3MgC), as the base structure. Possessing high physical and mechanical properties, these components ensure a composite powder mixture with high-quality new characteristics. Nevertheless, at the initial stage of testing, coatings made from them are not free from

Citation: Shchepetov VV, Fialko NM, Bys SS. Self-Lubricating Tribotechnical Nanocoatings Based on Magnesium Compounds. Japan J Res. 2026;7(5):190.

drawbacks, such as low toughness, increased brittleness, and a tendency toward embrittlement and chipping. At the same time the additional introduction of functional titanium (Ti) and chromium (Cr) master alloys into the initial powder mixture resolves the tribological issue associated with low crack resistance. The material remains hard but loses its brittleness.

The further formation of high-quality coatings involved the use of advanced technologies, including mechanochemical synthesis (MCS) and detonation-gas spraying (DGS). Structurally free magnesium carbide (MgC_2) was added to the resulting powder nanocomposition, and the mechanical mixture thus formed was stirred until a uniform distribution of components was achieved, making it ready for detonation-gas spraying.

The obtained cermet composite nanopowders were deposited via the detonation-gas method onto prepared steel 65G substrates (HRC 62−65). To improve adhesion strength, a classic heat-resistant bond coat of the $NiCrAl_Y$ system was preliminarily applied to the working surface of the samples. The adhesion strength, determined by the pin method, was approximately 115 MPa for the magnesium-containing coatings, with a porosity of no more than 0.5%. After grinding, their initial roughness was Ra 0.32−0.63 μm.

Testing of the sprayed samples was performed using a face-to-face scheme under distributed contact conditions at room temperature in a continuous sliding mode (from 0.1 to 4.0 m/s) under a constant load of 12.0 MPa. The environmental impact, sliding speeds, and loads were selected to bring the processes of physical-chemical mechanics of friction as close as possible to real operating conditions. The research program also included a comparative analysis of the friction parameters of the developed coatings with similar tribological values for tungsten-containing coatings of the WK15 type and widely used coatings made of alloyed nichrome [5].

The structure of the coatings was studied at all levels by examining the "technology–structure" and "constitution–properties" relationships based on a complex of modern physical and chemical methods of structural-phase analysis, which included examining the surface layer zones at macroscopic and microscopic scales. The comprehensive research methodology included metallography (a "Neophot-32" optical microscope with an attachment); the particle size of the powder mixtures was measured using an ANALYSETTE 22 Tec. plus instrument; durometric analysis was performed on a LECO M-400 hardness tester; scanning electron microscopy was conducted using a JSM-840 SEM; and X-ray diffraction phase analysis was carried out on a DRON-UM1 diffractometer.

Research Results and Discussion

An analysis of the causes of the main processes on friction surfaces indicates a relationship between external mechanical factors and the plastic deformation process, which dictates the sequence and qualitative patterns of friction and wear phenomena and determines the dominant wear mechanism.

The wear patterns of the investigated coatings are presented as graphs of averaged values of wear intensities and friction coefficients obtained during tests under monotonically increasing sliding velocities and a constant load of 12.0 MPa (Fig. 1). It can be stated that throughout the entire test range, the monitored tribological parameters of the magnesium-containing coatings are minimal, stable, and steady compared to the reference coatings, which ensures their normal mechanochemical (oxidative) wear process.

Research on magnesium-containing cermets based on complex ternary compounds has been ongoing for a long time; however, their tribological capabilities, despite individual achievements, remain far from the desired results [6].

As a result of electron probe microanalysis (EPMA) performed on a "Camebax SX" system, it was established that the investigated magnesium-containing nanocoating was obtained as a complex cermet multi-phase dispersed structure based on a high-strength, heat-resistant aluminum-magnesium spinel matrix ($MgAl_2O_4$) and discrete nanoscale reinforcing phases, such as titanium carbide (TiC), silicon carbide (SiC), chromium carbide (Cr_3C_2), as well as silicides (TiSi, $TiSi_2$, CrSi, Cr_2Si) and intermetallic compounds of the TiAl, $TiAl_3$, CrAl, and $CrAl_4$ types. Chemically inert carbides, silicides, and intermetallics distributed in the matrix structure serve as highly effective reinforcing phases, forming a composite nanocoating with discrete strengthening. Furthermore, the properties of the composite magnesium-containing coating are characterized by a combination of high microhardness and a low friction coefficient. Thus, the integral microhardness ranges within 12–15 GPa. Taking into account the phase intensity distribution

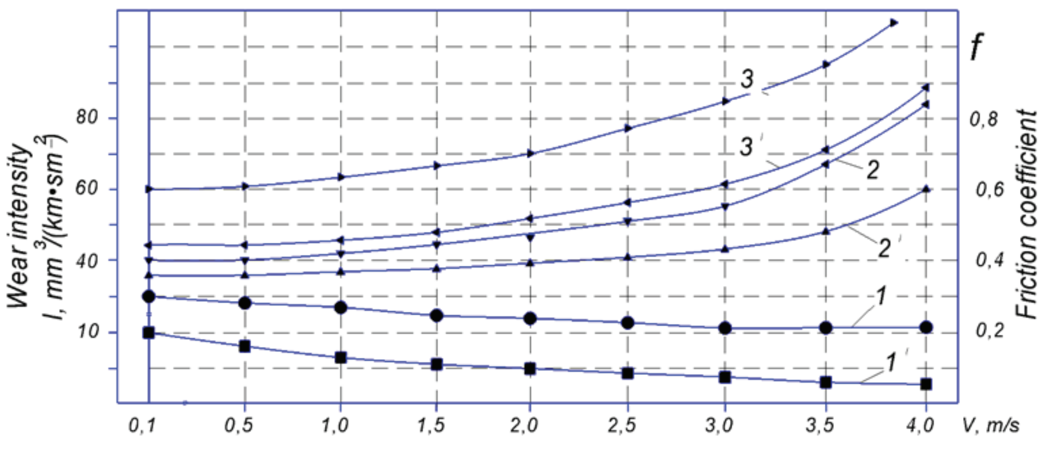

***Fig. 1** – Comparative dependencies of wear intensities (1, 2, 3) and friction coefficients (1', 2', 3') of magnesium-containing coatings (1, 1'), WK15-type coatings (2, 2'), and alloyed nichrome (3, 3') at P = 12.0 MPa.*

profile and their real stoichiometry, it can be assumed that the balanced quasi-chemical equation of interaction of the initial components is as follows:

$$3Mg_2SiO_4+4Al_3MgC+3Ti+Cr+6{,}5O_2 \rightarrow 5MgAl_2O_4+2TiC+3SiC+TiAl+CrAl+5MgO.$$

The O_2 missing in the right-hand side, in our opinion, is compensated for by the endogenous oxygen of the forsterite and the atmospheric environment under spraying conditions.

The reinforcing nanodispersed phases, uniformly distributed within the coating, exert a decisive influence on the dislocation mechanism. Figure 2 presents a typical dislocation grouping in the form of a cellular structure consisting of relatively closely packed dislocations with areas of dislocation loops located across almost the entire matrix. This leads to a density gradient and, presumably, reinforcing formations in areas with high dislocation density.

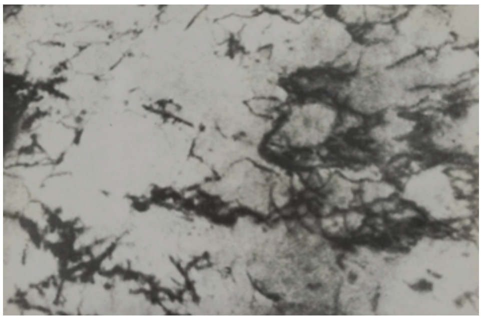

Fig. 2 *– Micrograph of the dislocation structure of the magnesium-containing coating (×20000).*

The friction and wear parameters of the studied coatings are determined not only by the structure and phase composition; the quality of the resulting surface structures, in which structural-thermal activation processes occur, also exerts a great influence. To investigate the microstructure of the surface layer, X-ray photoelectron spectroscopy (XPS) was applied.

The obtained results allowed us to generalize that the passivating surface films have an ultra-dispersed structure and consist of a conglomerate of components that belong to the composition and have interacted with atmospheric oxygen. According to the stoichiometric composition, they represent a complex mixture of fine oxides (SiO_2, C_2O_3, MgO, Al_2O_3) and the presence of spinel phases, mainly $MgAl_2O_4$, $MgCr_2O_4$, $MgTiO_3$. The presence of complex ternary compounds resembling MgO,Al_2O_3, TiO_2 is also not excluded. It was established that an orthorhombic crystal system closes to the sillimanite structure, most likely low-temperature mullite, was obtained reactively from solid solutions of Al_2O_3 and SiO_2. In addition, a minor presence of cordierite ($Mg_2Al_4Si_5O_{18}$) is possible. However, it was not possible to detect the cooperation products of chromium and aluminum, although activation under friction loading raises the threshold of their active interaction.

It should be clarified that the formed highly dispersed heterogeneous passivating oxide film stands out for its significant plasticity and hardness, high wear resistance, increased thermal stability, and corrosion resistance at low values of shear resistance. It should be noted that the processes of formation and destruction of surface oxide structures are in dynamic equilibrium and are automatically self-regulated, which is driven by the phenomenon of structural adaptability under steady mechanochemical wear conditions. Thermodynamic analysis and experimental studies of the frictional interaction of nanocoatings against the background of the formation of complex oxide structures allowed us to establish a relationship between wear patterns and the energy characteristics of the formation of metastable carbides capable of generating a self-renewing antifriction graphite layer. The results showed that for these test modes, the specific wear work value, amounting to about 10^4 kJ/mm^3, is a necessary and sufficient energy condition for the solid-phase decomposition of metastable magnesium carbides (MgC_2) contained in the nanocoating. This decomposition yields surface carbide α-graphite, serving as a source for a self-reproducing antifriction graphite layer that regenerates according to the solid-phase sequence: $MgC_2 \rightarrow Mg_2C_3 \rightarrow C$

We note that under thermodynamic exposure, the presence of Si as a catalyst in the structure stimulates graphitization. The decomposition temperature of metastable carbides depends, in particular, on the initial particle size. As established in these tests, due to high surface energy, the temperature at which decomposition begins in the areas of actual contact for nanoparticles is 200−300∘C. Figure 3 shows the topographies of the friction surfaces obtained at sliding velocities *corresponding* to running-in (V_1= 0.12 m/s) and the onset of normal wear (V_2=1.2 m/s).

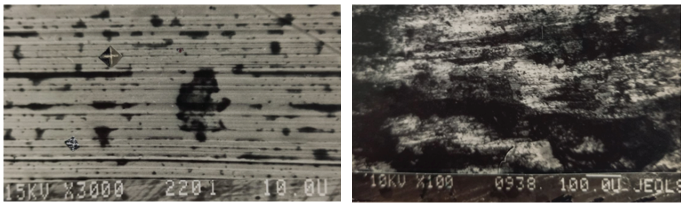

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Fig. 3 *– Topography of friction surfaces under graphitization conditions at V_1= 0.12 m/s (a) and V_2 =1.2 m/s (b).*

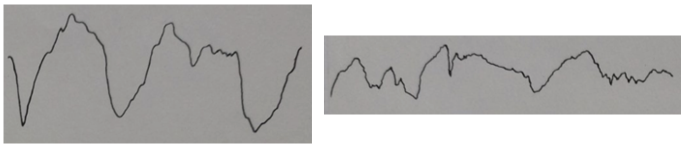

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Fig. 4 *– Profilograms of the friction surfaces of the magnesium-containing coating: a) under running-in conditions; b) during normal mechanochemical wear (Vertical Magnification ×1000; Horizontal Magnification ×40).*

It can be seen that at v_2, the antifriction graphite layer covers most of the working surface, ensuring an increase in the actual contact area and a decrease in the specific load due to an increase in the bearing length. This occurs because the graphite microparticles fill and level out micro-irregularities, anchoring themselves in micro-valleys.

The profilograms of the friction surfaces are presented in Fig. 4. Their examination allows us to conclude that during sliding friction, the topography is determined by the dominant type of wear and indicates the formation of an equilibrium roughness, which is optimal for specific friction conditions, ensuring minimal wear.

The main factors determining the qualitative level of thermomechanical carbide graphitization include the degree of structural dispersion, specific pressure, operating temperature, contact zone temperature, the presence of elements initiating decomposition, environmental influences, and factors related to material composition, structure, defects, and others.

For a comprehensive analysis of the quality of the surface structure where activation processes occur, an additional study was conducted using transmission electron microscopy (TEM). Figure 5 shows a typical friction surface of the magnesium-containing coating and its electron diffraction pattern from an area of about 0.5 μm^2, capturing changes in the fine structure.

The electron microscopic study under friction loading revealed that an ultra-dispersed structure with a fragment size approximately 20–35 nm forms in a thin surface layer (~10−25 nm). A spot pattern of reflections is clearly visible, forming ring structures that testify to its polycrystalline state, representing an oriented mixture of highly dispersed phases. The emergence of the highly dispersed structure indicates that plastic deformation of the surface layer is most likely carried out via a rotational mechanism due to the relative sliding of its fragments.

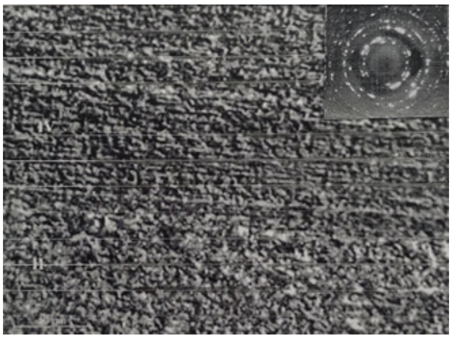

Fig. 5 *– Microscopic image of the local microvolume structure of the surface layer and a micro-electron diffraction pattern of the composite coating based on magnesium compounds (structure at a depth of 2–3 μm).*

At the same time, the low and stable values of both friction coefficients and wear intensities (see Fig. 1, curves 1^1 and 2^1) are due to the formation of an integral, self-regulating conglomerate of finely dispersed surface structures, which are mechanical mixtures of oxide compounds with low shear resistance. It should be noted that the composition of self-regulating dynamic structures may vary within certain limits, but their overall presence under conditions of dynamic formation and destruction remains unchanged.

Furthermore, they screen the adhesion-molecular interaction and, under local contact pressures and temperatures, form dense surface films free of cracks and chips. These films act as a barrier to oxygen diffusion, helping not only to reduce the oxidation rate and increase heat resistance but also actively performing the role of solid lubricants. Thus, during friction, the investigated magnesium-containing coating forms a stable surface dynamic state of self-originating and self-regulating ordered structures possessing a stable self-lubricating property. Their self-lubricating capacity is provided by a complex heterogeneous oxide-graphite surface film formed by the additive interaction of three structural components:

1. Components of the magnesium base structure, which possesses a high passivating ability;
2. Layered structural surface oxides having low shear resistance;
3. The presence of effective structurally free magnesium carbide, which serves as a source for surface graphitization.

The regenerated complex of self-lubricating heterogeneous structures under friction conditions possesses an effective capacity for self-healing and self-regulation. Its actual operation ensures a steady manifestation of structural adaptability, providing stable and reliable minimization of friction and wear parameters.

The operational assessment of the nanocoating quality is linked to the technological optimization of detonation-gas spraying modes. To obtain high-quality nanocoatings, the problem of analyzing an adequate mathematical model was solved, taking into account a complex of technological and operational factors and their impact on the optimization process. Optimal spraying regimes were implemented for parameters such as particle size distribution and loading depth, barrel filling degree, working gas ratio, spraying distance, justification of the carrier gas, and detonation frequency (number of shots per second), etc. [7].

Thus, by controlling the optimal technological process of forming nanocoatings based on magnesium compounds, it was possible to reproduce not only the intended chemical composition but also to obtain a predicted structure possessing an optimal complex of properties that ensure the stability of the structural adaptability phenomenon. Concurrently, reliable quality in the strength and plastic properties of the nanocoatings was achieved, with variations within a single batch of samples remaining stable at 5–10%.

As can be seen from the comparative research results (Fig. 1), the reference coatings are significantly inferior in terms of tribological characteristics compared to the self-lubricating nanocoatings based on magnesium compounds developed by the authors. Thus, detonation nanocoatings based on magnesium-containing compounds possess high antifriction properties and vastly surpass widely used tungsten-containing tribological materials. As promising coatings, they feature significant adhesion strength and an ability to provide high antifriction behavior through their composition, which, along with self-lubricating properties, opens up possibilities for their application in minimizing wear.

Taking into account the results obtained in papers [8, 9], the developed composite magnesium-containing nanocoatings can be considered for increasing the reliability and operational resource of parts operating under extreme conditions. Their application is justified for improving efficiency at repair enterprises when restoring worn components, which will significantly increase their wear resistance and solve the problem of restoring previously non-repairable parts [10]. As test results indicate, the proposed coatings ensure the operational reliability of tribological joints in accordance with the conditions imposed on new competitive materials obtained by the detonation method [11]. The developed magnesium-containing composite nanocoatings can be considered an alternative to promising materials for service in modern machinery units, including aerospace applications.

Studies on the friction and wear patterns of magnesium-containing composite nanocoatings demonstrate the feasibility of their application to increase the operational reliability and durability of friction parts. It can already be stated that their practical implementation will improve reliability and technical lifespan, and reduce repair costs in the event of operational damage. Components subject to strengthening and restoration include compressor and fan blades, rotor supports, bearing units, crankshafts, seats, disk and oval surfaces, cam mechanisms, sliding bearings, reciprocating pairs, and high-speed, heavily loaded units where the use of traditional lubricants is undesirable.

The presented work is motivated by a growing practical interest in producing coatings from new materials with a controlled phase composition and a functional self-lubricating property. The results stimulate the pursuit of creating new magnesium-containing compositions and open up prospects for developing technologies to obtain scientifically substantiated antifriction self-lubricating coatings. It should be noted that the proposed powder mixture based on magnesium reagents for forming antifriction self-lubricating magnesium-containing coatings can be applied by various technological methods that utilize powder materials.

The development and testing of magnesium coatings, despite the economic difficulties experienced by the country, is a necessary component of the technical and social development of both science and society as a whole.

Conclusions

1. A composite magnesium nanocoating based on a mechanical mixture of forsterite and a triple compound of magnesium-trialuminum carbide has been developed and tested. It is characterized by stable and low friction and wear parameters across various sliding velocities under a load of 12.0 MPa. The investigated nanocoatings exhibit a wear resistance 3.3 to 8.5 times higher than traditional tungsten-containing and nichrome coatings.
2. It is noted that the quality of the sprayed nanocoating is inextricably linked to solving the reproducibility problem of the technological process. Through experimental research, the optimal technological regime for spraying the composite magnesium-containing powder was refined. During nanocoating formation, this regime ensured not only the necessary chemical composition but also the creation of a stable antifriction structure. Stability in the properties of the nanocoatings was achieved, with variations during spraying amounting to less than 10%.
3. By optimizing the technological regime, a detonation magnesium-containing nanocoating was obtained. The minimization of its wear intensity and friction coefficients is driven by the formation of a self-regulating structured layer, which, under friction conditions, is governed by a cooperative action associated with self-organization. This self-organization establishes a dynamic equilibrium through the additive interaction of graphite released from carbides, complex oxide structures, and the passivating structures of the matrix phase.
4. The phase composition of the magnesium-containing nanocoatings was investigated using physical analysis methods, revealing cermet multi-phase fine-grained formations based on aluminum-magnesium spinel and discrete nanoscale reinforcing phases. The structure of the passivating surface films was determined, which possess increased plasticity and hardness, high wear resistance, elevated thermal stability, and corrosion resistance under low shear resistance.
5. It was established that the transition to an ultra-dispersed surface structure (grain size 20–35 nm) activates a rotational deformation mechanism, providing high integral microhardness (12.0–15.0 GPa) coupled with low shear resistance. The physical mechanism and the main factors determining the level of thermomechanical carbide graphitization were examined.
6. The developed self-lubricating composite nanocoating based on magnesium compounds of forsterite and magnesium-trialuminum carbide expands the achievements of modern tribological materials science. The investigated composition, capable of self-lubricating, can be used both for strengthening and for the high-quality restoration of worn parts using various surface engineering technologies that employ powder materials..

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