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The Influence of Filler Dispersion and Gradient Structure on the Tribological Properties of Composite Coatings

M. V. Kindrachuk, A. O. Kornienko, V. V. Kharchenko,
P. D. Stukhlyak*, M. A. Hlovyn, and I. V. Kostetskyi

*State University 'Kyiv Aviation Institute',
1 Liubomyr Huzar Ave.,
UA-03058 Kyiv, Ukraine*

**Ternopil Ivan Puluj National Technical University,
56 Ruska Str.,
UA-46001 Ternopil, Ukraine*

The stress–strain state of a material loaded by friction forces has a significant impact on the wear resistance. The value of stresses and their distribution in the coating determine the coating resistance to wear. According to the results of this research, a positive effect of the gradient on-base layer on the wear resistance of multilayer coatings is established; it is dependent on the stress–strain state of the composition, the distribution of stresses, and their damping properties. Herein, preference should be given to the order of arrangement of its layers by sizes of inclusions from-coarse-to-fine ones. With such a structure, the wear resistance of the coating will be the highest in given friction conditions.

Key words: gradient composite coatings, matrix, filler, stress–strain state, wear resistance.

Напружено-деформований стан матеріалу, навантаженого силами тертя, має істотний вплив на зносостійкість. Величина напружень та розподіл їх у покритті визначають стійкість покриття проти спрацювання. За резуль-

Corresponding author: Volodymyr Volodymyrovych Kharchenko
E-mail: vvkhna@gmail.com

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татами досліджень виявлено позитивний вплив ґрадієнтного підшару на зносостійкість багатошарових покриттів, що зумовлено напружено-деформованим станом композиції, розподілом напружень, їхніми демпфувальними властивостями; водночас перевагу слід віддавати порядку розміщення шарів із вкрапленнями у підшарі від великих до дрібних. За такої будови зносостійкість покриття буде найвищою для цих умов тертя.

Ключові слова: ґрадієнтні композиційні покриття, матриця, наповнювач, напружено-деформований стан, зносостійкість.

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1. INTRODUCTION

The Essence of the Problem and Its Connection with Practical Tasks.

The work experience and the results of previous numerous studies indicate that in most cases, the functional properties and the resource of various types of machines and equipment are determined by intensive wear of heavily loaded parts of friction units and working bodies. At the same time, traditional methods for the deposition of coatings do not meet the constantly increasing requirements for the performance of critical friction parts of mechanisms in conditions of high dynamic loads, high temperatures, active corrosive media, and the presence of abrasive flows.

An effective way out of the current situation is the formation of wear-resistant gradient layers on of a heterogeneous composition and layered or discrete structure of the matrix-filled or skeletal type on the surface of parts made of structural materials.

The peculiarity of gradient composite coatings is that the coating itself is a powder matter, or a powder medium was used to obtain it as a source of elements or compounds that form the coating. Thus, gas thermal coatings (GTC) are a powder pseudo-alloy of sprayed powder particles; composite electrolytic coatings (CECs) are a mechanical mixture consisting of a metal matrix and powder filler; eutectic coatings (EC) are a skeletal-type formation with a reinforcing or restoring powder layer melted by heat treatment on the surface being protected.

Despite the large amount of information on individual problems related to the obtained GTC, CEC, and EP, today, there is no systematic information on the combined method for formation of gradient layers. As for the tribological and tribometrical characteristics of multilayer coatings with different gradients in the layers, formed, in particular, with hard inclusions of different sizes and different volume contents, they are insufficient for generalization, or they are obtained using individual, mostly unsuitable for comparison, methods. Therefore, there is a timely and urgent need to study and systematize the tribological characteristics of nickel-based CECs, containing carbides, nitrides, and borides (TiC,

SiC, TiN, CrB₂, and TiB₂) as a strengthening phase. Actually, due to the structural differences in the composite layers, they provide the treated surface with the necessary properties, usually sharply different in terms of structural gradient from the properties of the matrix material or filler of the coating itself, as well as the base on which it is deposited.

Review of publications and analysis of unsolved problems. Gradient layers are composite matrix-filled or reinforced coatings, whose regular structure is disturbed in the gradient direction (that is, there are differences in the concentrations of elements in the matrix alloy, between the matrix and inclusions, or in the shape, number, and size of the inclusions, as well as in the distance between them, changing by certain laws).

Gradient coatings can be modelled by a set of single-layer composites with different laws of gradient change. The heterogeneity of the structure of multilayer coatings causes a wide range of new research tasks that do not arise during the study of the tribological behaviour of traditional materials, and to a lesser extent, single-layer compositions. Such tasks, in particular, include the problem of determining a rational gradient of properties and the tribometrical characteristics of coatings.

Single-layer coatings of the matrix-filled or skeletal type significantly improve the performance of heavily loaded friction units of machines and mechanisms, in particular, they can work in sliding bearings of cone drill bits [1–4]. Analysis of the antifriction properties of composite materials has shown that the size of the filler inclusions and their volume content play an important role [5]. The wear resistance of heterogeneous materials is significantly affected by the stress state that arises during contact with the conjugated surface [6].

In Reference [7], the results of the computational determination of the stress–strain state of composite materials with identically oriented columnar formations are presented. The relationships between the stress concentration, the mechanical properties of the matrix and inclusions, and the density of inclusions have been established. In the material structure, regions with an increased level of stress were identified as the most susceptible to the onset of destructive processes. The problem of the evolution of the structure and mechanical properties of composite coatings as a result of plastic deformation of surface layers during friction is considered in Ref. [8]. If during the friction process, the coating structure changes in such a way that the rule of a positive gradient of properties is valid, normal external friction takes place. This makes it possible to affect the parameters of the friction pair and the properties of the deposited layers basing on the known data on the determination of mechanical and strength properties as a means of controlling wear mechanisms. Using the model of a composite material including an annular transition zone between the filler and the matrix with a known law of the change in their mechanical properties, the nature of the stress state caused by loading with sliding friction forces has been studied in

Ref. [9]. It was shown that local stresses in such a composition, depending on the type of loading of an elementary volume, can be predicted and calculated, taking into account the mechanical properties and the structure of the transition zones formed due to the matrix–inclusion diffusion interaction according to the corresponding state diagram.

Thus, the available theoretical and experimental data about composite coatings, together with knowledge of the mechanisms of their formation, are an important prerequisite for the practical formation of composite layers, but not sufficient. Therefore, the aim of the work was to study the influence of the structure and composition of matrix-filled type compositions and the gradient of properties on the tribological behaviour of single- and multilayer coatings.

2. EXPERIMENTAL/THEORETICAL DETAILS

Research Methodology. Composite electrolytic coatings were formed using electrolytic nickel powder and the strengthening SiC phase of different dispersions. The coatings were deposited onto prismatic samples with a size of 10×10×5 mm. Coarse particles (fractions 100/80, 50/40, and 28/10 μm) were deposited on the horizontal cathode by the pulsed stirring of the electrolyte at a current density of 5–10 A/dm², pH 3–4, and electrolyte temperatures of 25 and 40°C. Coatings containing more dispersed particles (fraction 5/3 μm , SiCN nanoparticles \cong 50 nm) were obtained on the vertical cathode with the constant stirring of the electrolyte at a current density of 4–5 A/dm². Multilayer coatings were obtained by successive layer-by-layer deposition of precipitates containing different amounts of filler of different dispersion.

Testing of samples with coatings was carried out on an M-22M installation under dry friction conditions at sliding speeds of $V = 0.5$ m/s and loads $P = 20, 40, 60$ N. The counter-body was from hardened steel HRC 45. The mating scheme was shaft-plane. Friction path is $L = 1$ km. The amount of wear was estimated by the loss of sample mass and the amount of linear wear of the friction pair.

3. RESULTS AND DISCUSSION

Compositions with different sizes and volume content of inclusions were studied. The content of silicon carbide inclusions in the coating depended on their sizes. The results of the chemical analysis of CECs with different fractions of inclusions are given in Table 1. The number of inclusions in the coating could be easily controlled within the volume content from 0% to 50–70% by changing the relationship between mixing and sedimentation.

Tribological studies have shown that CECs with 28/20 inclusions and

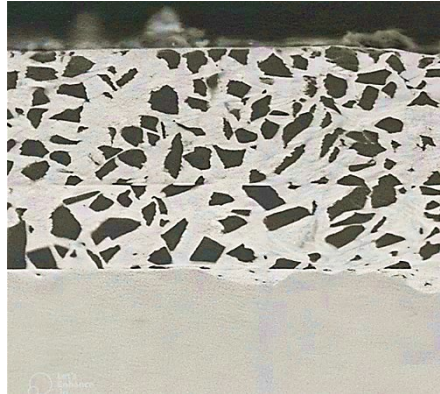
TABLE 1. Influence of the size of SiC inclusions on their content in CEC.

Size of inclusions, μm	Volume content in coatings, vol.%
50 nm	3.8
5/3	8.66
28/20	24.66
50/40	37.50
100/80	43.11

their volume content of 24% have the highest wear resistance. The coating microstructure is shown in Fig. 1. The results of the structure studies indicate that compositions with volume content of inclusions within 20–40% have an optimal wear resistance. The same results were obtained for CEC Ni–TiB₂ in Ref. [5]. However, the optimal composition of alloys characterized by maximum wear resistance was at 20–30% of inclusions.

To date, there is no explanation of such regularities from a single viewpoint. It has not been clarified which of the parameters—volume fraction, size of inclusions, or the smallest distance between them—determines certain tribological parameters and mechanical properties. Analysis of the obtained results found that for optimal compositions (20–30%, inclusion particle size 28/20), such regularity is observed: the ratio of the distance between the centres of inclusion particles L to their sizes d , L/d , is of about 3. Then, at a volume content of 24%, this intercentre distance is of 90 μm ; therefore, $L/d = 3.21$.

Since among the obtained coatings, only coatings with 28 μm inclusion particles had the optimal content, the compositions of other filler fractions with different volume contents were additionally studied. According to the results of tribological studies, the highest wear re-

**Fig. 1.** Microstructure of CEC Ni–SiC₂₈, $\times 100$.

sistance was exhibited by CECs with a volume content of filler of about 24%. The results of tribological studies of CECs with inclusions of different fractions of the same volume content of about 24% are shown in Fig. 2. As seen, the highest wear resistance was manifested by CECs with inclusions sizing within 30–50 μm . Note that, for inclusions of different sizes with a volume content of about 24%, the ratios of intercentre distances to diameters are of about 3 (Fig. 3).

These findings are consistent with the conclusions in Ref. [10], where the influence of the structural state of a composition on its stress–strain state and wear resistance was investigated by the polarization-optical method. As a result of the study of the factors determin-

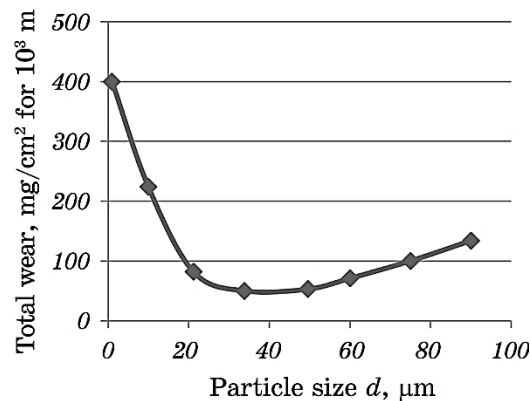


Fig. 2. Dependence of the wear resistance of Ni–SiC CECs on the size of SiC particles at a load of 20 N and friction speed of 0.5 m/s (fillers' volume content of 24%).

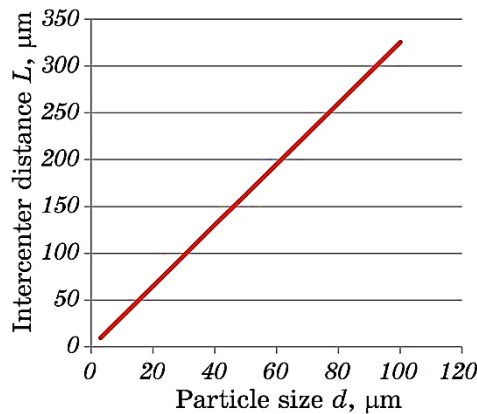


Fig. 3. Dependence of the distances between the centres of particles on their size at a volume content of 24%.

ing the location of the zones of maximum tangential stresses in the composite material, the conditions for optimizing their distribution that ensure maximum resistance to fatigue wear were established, and a quantitative assessment was given for the ratio of the intercentre distance to the particle size, under which these conditions are realized: $L/d \geq 3.3$.

Since the main role of solid particles is to strengthen the matrix, the presence of hard inclusions strengthens or reinforces the matrix, limiting its plastic flow. Probably, at this ratio ($L/d \geq 3.3$), the particles most effectively compress the matrix and limit its plastic deformation. In this case, the zone of increased tangential stresses, which arises due to the interaction of stress fields from neighbouring inclusions, deepens into the material below the inclusions. This, in turn, reduces the load on the matrix between the inclusions and provides a positive stress gradient from the friction surface. When the intercentre distances become smaller, the strength of the composition decreases due to the formation of cracks at the weakened particle–particle interfaces.

It is possible to predict the stress for the onset of plastic deformations $\langle \sigma_1 \rangle$ of composite material under friction depending on the nature, the volume ratio of its components, and the friction coefficient according to the analytical studies of the stress–strain state [8], using the formula

$$\langle \sigma_1 \rangle = \sqrt{2}(\sigma_T)_m / \sqrt{A + f^2 B},$$

where $(\sigma_T)_m$ is the yield strength of the matrix material; f is the coefficient of transformation of normal forces into tangential forces during friction, which with some assumptions can be considered the friction coefficient; A and B are coefficients, which depend on the mechanical characteristics and volume content of the components.

The relative minimum value of the average longitudinal compressive stress, $\langle \sigma_1 \rangle / \langle \sigma_T \rangle_m$, at which the process of plastic deformation starts, can be taken as the coefficient of plastic compression, which serves as a measure of the degree of strengthening. It was shown that, at a volume content of the filler $\xi < 10\%$, strengthening is not significant and $\langle \sigma_1 \rangle / \langle \sigma_T \rangle_m$ approaches 1. At a denser arrangement of particles, $20 < \xi < 40\%$, plastic deformation is significantly limited. When $f > 0.3$, the increasing volume content of the strengthening phase to 40% is not effective, since it does not affect the average normal stresses for the onset of plastic deformation. In addition, due to reducing distance between the particles ($\xi > 40\%$), significant local strengthening of the matrix occurs, which leads to a decrease in strength because of the formation of cracks in the weakened areas of the matrix between the particles.

It should be noted that the range of the filler volume content that provides the highest wear resistance of the composition, is influenced by the

physical and mechanical characteristics of the matrix and the filler. Thus, for the Ni–TiB₂ system, the efficient CEC performance is ensured at a filler volume content of 20–30%, while, for the Ni–SiC system, at 20–40%, which is probably related to the different relationships between the mechanical properties of the matrix and the filler. In composite materials where the mechanical characteristics of the filler and the matrix differ less, for example, in the N–SiC system (Fig. 4), the stress concentration decreases along with preserving the trend of its change with changing filler content. Calculations for determining the stress concentration were performed using the formula from Ref. [7]. The initial data for the calculation were for the Ni matrix as follow: elastic modulus $E_m = 1.86 \cdot 10^5$ MPa, shear modulus $G_m = 0.72 \cdot 10^5$ MPa, Poisson's ratio $\nu_m = 0.3$; for TiB₂ inclusions: $E_f = 5.1 \cdot 10^5$ MPa, $G_m = 2.3 \cdot 10^5$ MPa, $\nu_m = 0.1$; and for SiC inclusions: $E_f = 3.94 \cdot 10^5$ MPa, $G_m = 1.8 \cdot 10^5$ MPa, $\nu_m = 0.1$.

$$K_{1r} = 2 \frac{G_f / G_m}{\eta + (1 + \xi)G_f / G_m} \left[1 + 4S \frac{\xi^2}{\pi^2} \left(3 + 7 \frac{\xi^2}{\pi^2} \right) \right], \quad (1)$$

where $S = (G_f / G_m - 1) / (G_f / G_m + 1)$; η and ξ are the volume content of the matrix and inclusions, respectively.

Expression (1) stands for both the mechanical characteristics and the volume content of components. The dependences of the maximum shear stress concentration K_{1r} on the volume content of inclusions for the Ni–SiC and Ni–TiB₂ compositions are shown in Fig. 4.

The presented findings indicate that there is such a volume content of inclusions (20–40%), at which there occurs a minimum intensity of local tangential stresses in the matrix. The minimum wear also corresponds to the region of 20–40 vol.% of SiC and TiB₂ in the compositions. Along with other factors, this can be explained by the lowest

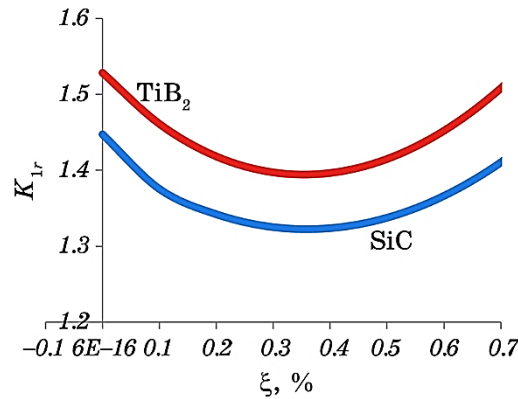


Fig. 4. Dependence of the longitudinal shear-stress concentration K_{1r} on the volume content of TiB₂ and SiC.

stress concentration.

As for the optimal size of inclusions (25–50 μm), it is probably related to the load they can take on. When their size is smaller or comparable to the size of the single contact spot (2–10 μm), this load is equal to the actual contact pressure. However, it is by 2–3 orders of magnitude smaller (equal to the contour load) for inclusions larger than 300 μm , when the ratio of the areas of the single contact spot and the solid inclusion coincides with the relative contact area [5]. Therefore, it is desirable to use solid inclusions with a size of more than 300 μm for more severe performance conditions, since, in this case, the fraction of the load taken by them is larger. In the transition region (10–300 μm), it can be expected that the fraction of the load on the solid inclusion will acquire some intermediate values. Therefore, the analysis of the macro- and microstructures of the friction surfaces of the CEC allows us to state that the relative contact area of the composition decreases with an increase in the size of the filler particles. For the composition containing particles with a size of 5 μm , smaller than the size of single contact spots, the main contribution to wear resistance is made by the Ni matrix.

Traces of setting plastic deformation are observed on the surface (Fig. 5, *a*). Figures 5, *c*, *d* show the friction surfaces of the CEC contain-

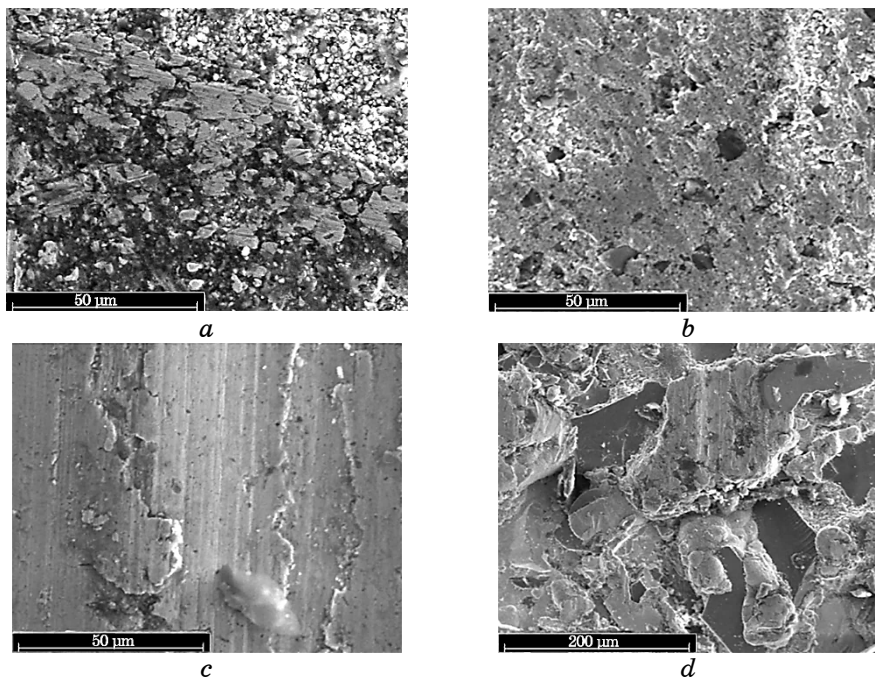


Fig. 5. Topographic images of friction surfaces of the studied CECs: Ni + SiC₅ (*a*), $\times 500$; Ni + SiC₂₈ (*b*), $\times 500$ (*b*); Ni + SiC₁₀₀, $\times 500$ (*c*); Ni + SiC₁₀₀, $\times 100$ (*d*).

ing filler with a size of $100\ \mu\text{m}$, when the ratio of the areas of the single contact spot and the area of the solid inclusion coincides with the relative contact area. On the friction surface, there are traces of abrasive wear by free or fixed (embedded in the counter-body) filler particles. For compositions with the optimal SiC particle size ($28\ \mu\text{m}$), the processes of seizure and abrasive or brittle fracture are non-characteristic; here a normal mechanical-oxidative wear process occurs (Fig. 5, *b*).

Since the stress-strain state depends on the coating structure, it was reasonable to study the influence of the structure gradient along the depth of the multilayer coating on its wear resistance. As known, in order to increase the wear resistance of machine parts, it is advisable to form wear-resistant gradient layers on the surface of parts made of structural materials, having a heterogeneous structure of the matrix-filled or the skeletal type [11–16].

Multilayer gradient coatings were obtained by successively forming layers, which had different sizes of inclusions. Two options for the arrangement of layers depending on the particle size of the filler were studied: 1) from course to fine inclusions, base $\rightarrow 100/80\ \mu\text{m} \rightarrow 28/20\ \mu\text{m} \rightarrow 5/3\ \mu\text{m} \rightarrow 50\ \text{nm}$ (Fig. 6, *a*); from fine to course inclusions: base $\rightarrow 5/3\ \mu\text{m} \rightarrow 28/20\ \mu\text{m} \rightarrow 100/80\ \mu\text{m} \rightarrow 50\ \text{nm}$ (Fig. 6, *b*).

Tribological studies of multilayer coatings showed that their wear resistance is higher than that of single-layer ones (Table 2).

Thus, single-layer coatings with nanoparticles are characterized by

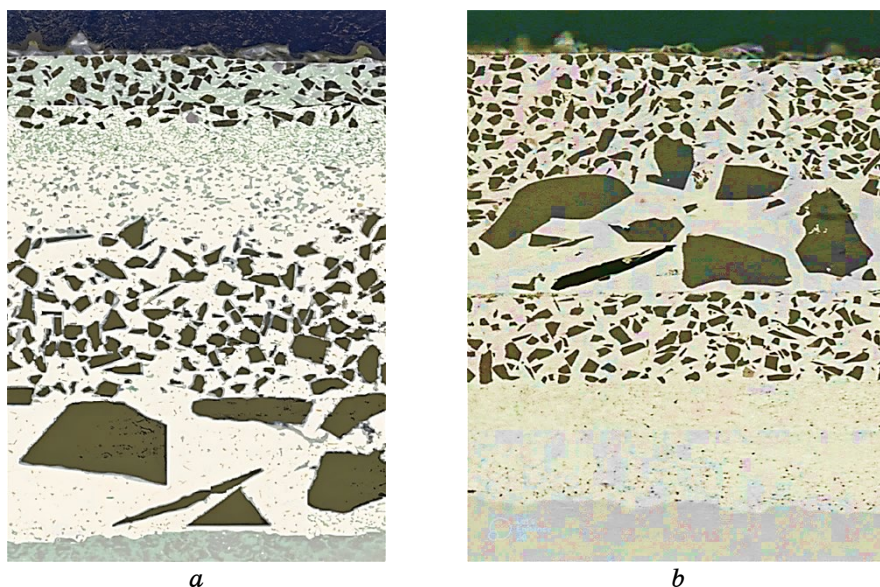


Fig. 6. Microstructure of multilayer gradient CEC containing SiC inclusions in a Ni matrix, $\times 100$.

TABLE 2. Results of friction and wear tests of coatings with fillers of different sizes.

Filler	Load, N	Friction coefficient	Wear, mg/km	Liner wear of friction pair, $\mu\text{m}/\text{km}$
SiCN ₅₀	20	1.3	32.4	47
	40	0.8	33.9	48
	60	0.7	36.6	50
Course → fine → nano	20	1.15	3.8	40
	40	0.82	9.3	49
	60	0.75	6.2	38
Fine → course → nano	20	1.3	4.0	48
	40	0.91	9.8	55
	60	0.75	6.5	45
SiC ₂₈	20	1.1	2.7	11
	40	0.9	4.4	22
	60	0.73	9.0	30
Course → fine → 28	20	1.52	1.8	16
	40	0.86	3.2	20
	60	0.84	2.1	27
Fine → course → 28	20	1.36	2.5	18
	40	0.85	4.0	27
	60	0.82	7.8	29

low wear resistance. However, if the coating with nanoparticles was applied to a gradient on-base layer, its wear resistance increased by an order of magnitude. At the same time, coatings with the order of layers arrangement by coarse-to-fine inclusions have greater wear resistance. This can be explained by the increase in the dissipative properties of such a composite, due to the sequence scheme from the base to the coating layers in the order of decreasing plastic and increasing elastic characteristics of their materials [17]. When a gradient coating is used as an on-base layer for a composition with the optimal size of inclusions, an increase in the wear resistance of the coating can be observed in the entire range of loads in the case of placing layers with inclusions from coarse to fine, whereas coatings with the order of layer arrangement from fine to coarse inclusions have somewhat greater wear.

4. CONCLUSION

The studies have established that the stress–strain state of the material loaded by friction forces has a significant impact on the wear resistance of the CEC. The value of the stresses and their distribution in

the coating determine the coating resistance to wear.

It has been established that the optimal volume content of the strengthening phase in terms of wear resistance is within 25–40%, which is related to the ratios of the physical and mechanical properties of the filler and the matrix. Such a volume content corresponds to the ratio of the intercentre distance of the particles L to their size d $L/d \gg 3$. This ratio imposes significant restrictions on plastic deformation and, as a result, strengthens the matrix. At $L/d < 3$, a significant local strengthening of the matrix occurs due to a decrease in the distance between the particles ($\xi > 40\%$), which leads to a decrease in the strength of the composition as a whole because of the formation of cracks at the weakened particle-particle interface. When $L/d \gg 3$, such strengthening is not significant, and $\langle \sigma_1 \rangle / \langle \sigma_T \rangle_m$ approaches 1.

The positive influence of the gradient on-base layer on the wear resistance of multilayer coatings has been revealed, which is related to the stress–strain state of the composition, the distribution of stresses, and their damping properties. Herein, preference should be given to the order of arrangement of its layers by the sizes of inclusions from coarse-to-fine ones. With such a structure, the wear resistance of the whole coating will be the highest in given friction conditions.

AUTHORS' CONTRIBUTIONS

Myroslav Vasyliovych Kindrachuk supervised the project and wrote the manuscript with contributions from all authors. Anatoliy Kornienko applied the coatings and investigated the physical and mechanical properties. Volodymyr Volodymyrovych Kharchenko conducted research on the tribological properties and worked on the manuscript. Petro Danylovych Stukhlyak developed conceptual ideas and provided critical revisions. Mykhailo Andriyovych Hlovyn analysed the literature and participated in conducting experiments. Ivan Volodymyrovych Kostetskyi participated in conducting the experiments.

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