

Досліджено структуру композитів на основі системи NbC з мідною зв'язкою, отриманих шляхом просочування металевим розплавом пористих NbC карбідних каркасів у вакуумі. З метою отримання пористого каркасу порошок NbC із середніми розміром ~ 1 мкм замішували на 5 %-ному розчині каучуку в бензині. Після сушіння суміш перетирали на ситі у гранули, які пресували у брикети розмірами $55 \times 30 \times 10$ мм. Для забезпечення інтенсифікації процесу та змочуваності просочування проводилось за температури 1400 °C. У результаті було отримано матеріал із дрібнозернистою двофазовою структурою.

Дослідження мікроструктури проводили методом скануючої електронної мікроскопії (SEM), хімічного складу – методом енергодисперсійного аналізу (EDS).

Твердість вимірювали за Роквеллом (шкала C), тріщиностійкість за непрямим методом Еванса-Чарльза.

Структура композиту складається із округлих зерен NbC, які утворюють неперервний скелет та прошарків мідної зв'язки. Середній розмір зерен та міжзернових прошарків зв'язки становить $1,8$ мкм та $1,1$ мкм, відповідно.

Аналіз зони взаємодії між NbC та Si методом EDS дозволив встановити присутність дифузійної зони товщиною $0,5$ мкм, яка утворюється внаслідок перерозподілу Nb та Si шляхом обмеженої розчинності. Наявність дифузійної зони дозволяє забезпечити міцний зв'язок між фазами та, відповідно, високий рівень механічних властивостей. Твердість та тріщиностійкість отриманого матеріалу становлять 40 HRC та 24 МПа \times м $^{1/2}$, відповідно.

Враховуючи фазовий склад та властивості розробленого композиту, його рекомендується застосовувати як альтернативний до композитів системи WC-Si у вигляді монолітного матеріалу або покриттів. Покриття наносили методом електроіскрового легування із використанням ручної установки МП-ЭЛ2. Товщина покриття становить 30 мкм, мікротвердість ~ 500 МПа, а коефіцієнт тертя по сталі без змащування $0,04$.

Розроблені матеріали рекомендуються для застосування в парах тертя у вигляді монолітного матеріалу або покриттів антифрикційного призначення

Ключові слова: кераміко-металічні матеріали, матрично-армована структура, триботехнічні характеристики, електроіскрове легування, антифрикційні покриття

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DEVELOPMENT OF THE COMPOSITE MATERIAL AND COATINGS BASED ON NIOBIUM CARBIDE

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1. Introduction

The combination of high hardness and wear resistance of refractory compounds of transition metals from group IV–VI in the periodic system of elements, and copper, in a

monolithic material, makes it possible to obtain composites and coatings with high thermal- and electrical conductivity. Using such materials is promising in order to apply coatings with specialized physical-mechanical characteristics, as well as to improve wear resistance of the tribojunctions' surfaces

that operate under extreme conditions [1]. The most common among such materials are the composites of the WC–Cu system [2]. Extending the range of ceramic materials for the composites with a copper bonding is significantly hindered by the low wetting capacity of copper and conventional copper alloys. To improve wetting capacity, it is a promising direction to dope copper with highly-active metals Zr, Ti, etc., or to increase the temperature of impregnation. The disadvantage of copper doping by highly-active metals is that even a small amount of impurities (~5 %), required to lower the contact wetting angle, significantly reduces the heat- and electrical conductivity of copper. Thus, it is more rational to perform the process of copper impregnation at temperatures that are 200–400 °C above the melting point of copper, as well as to use the thermodynamically stable super-hard refractory compounds as components.

A promising material to be used in the form of a component in the composites with a copper matrix is, in particular, niobium monocarbide. Its density (7.8 g/cm³) is almost twice lower than the density of tungsten carbide (15.8 g/cm³) while the cost is only 30 % higher. Therefore, it is a relevant task in materials science to develop composites with a copper matrix based on niobium carbide.

2. Literature review and problem statement

At present, scientists are searching for new ceramic phases that are compatible with copper, as well as for the new production techniques. Among them, boron carbide has been investigated [3], in the amount of up to 3 % by weight, when obtaining the composites using the method of solid-phase sintering of the pre-formed billets; higher chromium carbide (Cr₃C₂) [4], in the system of titanium diboride (TiB₂) – copper, under conditions of its reaction thermal synthesis [5]. Paper [6] suggests obtaining the composites that were strengthened with tungsten carbides by applying the method of centrifugal reinforcement, or surfacing [7]. An analysis of results reported in papers [3–7] reveals that a general drawback of composites is their high (~10 %) residual porosity. This is predetermined by that such super-hard compounds as B₄C, TiB₂, TiC and others, which have been explored lately, are poorly wetted by molten copper at temperatures close to the melting temperature of copper [8]. In addition, reported technologies for obtaining composite materials do not make it possible to achieve a high concentration of refractory compounds (>50 % by volume) in a composite. Therefore, in order to obtain composites with a copper matrix, it is rational to apply a method for the impregnation of porous carbide skeletons with molten [9]. This method allows obtaining almost pore-free materials containing refractory compounds in the range of 60–80 % by volume. However, when applying the method of impregnation, there is an issue related to wetting the refractory phase with a molten. Because the compounds examined in papers [3–7] are poorly wetted by copper, there is a need to find the new refractory compounds for composites with a copper matrix.

A promising material for obtaining composites with a copper bonding using a method for the impregnation of porous ceramic skeletons with molten is, in particular, niobium monocarbide (NbC), which is well wetted by metals [10] and is characterized by high hardness and chemical stability [11]. Using NbC as a component of composites with a copper matrix, obtained via impregnation, makes it possible to elim-

inate the disadvantages characteristic of composites, which were described in papers [3–7]. In other words, to ensure the absence of residual porosity and to increase the content of the carbide phase of up to 60 % by volume.

3. The aim and objectives of the study

The aim of this study was to establish regularities in the formation of structure of composites in the system NbC–Cu when obtaining materials using a method of the impregnation of porous carbide skeletons, and to define their physical-mechanical characteristics. That will make it possible to construct the wear-resistant composite materials, as well as coatings for antifriction purposes.

To accomplish the aim, the following tasks have been set:

- to devise a technology for obtaining composites of the system NbC–Cu applying a method of impregnation of porous carbide skeletons with molten;
- to establish the character of structure formation, the phase composition, and properties of composites of the system NbC–Cu;
- to devise a technology for applying coatings from composites of the system NbC–Cu using a method of electric-spark doping;
- to explore the morphology of electric-spark coatings from composites of the system NbC–Cu and to define their tribotechnical characteristics.

4. Materials and methods to study the process of obtaining the NbC–Cu composite, as well as coatings based on it

The prerequisite for obtaining composites with a matrix based on copper is, first of all, the capacity of the refractory phase to be wetted with a copper melt. In order to use composites with a copper matrix as the carbide phase, there are the carbides of transition metals from group IV–VI in the periodic system of elements, such as V, Cr, Nb and Ti. A comparative analysis reveals (Fig. 1) that the carbides of Cr and V, which are wetted relatively well with the molten copper (contact wetting angle $\Theta < 90^\circ$), are characterized by low microhardness while the carbide of Ti, having high microhardness, is not wetted ($\Theta > 90^\circ$). In terms of wetting capacity and microhardness, the carbide of Nb accepts intermediate values, which is why it is the most promising one, among the examined refractory compounds, for the reinforcement of copper matrix.

The source materials used included the powder of NbC (TU 6-09-03-75); the composition of the carbide applied (Nb≥88.5 %, C≥11.1 %) was in line with formula NbC_{0.98}; the average particle size was ~1 μm; and copper of grade M0b in line with GOST 859-2001 (analog of Cu–OF (CW008A)). The samples were fabricated by the method of carbide skeleton impregnation. In order to make a porous skeleton, the carbide powder was plasticized by mixing on a 5 % solution of rubber in gasoline. After drying and granulation, the powder was pressed in a steel mould at a pressure of ~500 MPa. Thus, we obtained the workpieces the size of 55×30×10 mm. The open porosity, measured using the method of weighing the moulded workpieces, amounted to ~40 %. To conduct the impregnation process, we have chosen a “top-down” method. In order to implement it, we placed, atop the

workpieces, the briquettes of copper calculated to fill 100 % of pores. Next, the workpieces were put in crucibles made of aluminum oxide with zirconium dioxide backfill. Next, the crucibles were placed in the vacuum furnace SShV-1.25/25-11, where we heated them in a vacuum under the mode that implies the stages shown in Fig. 2: I – slow heating up to 1,000 °C in order to remove the plasticizer without destroying the workpiece; II – rapid heating up to 1,400 °C; isothermal aging at 1,400 °C, during which capillaries of the porous carbide skeleton are filled with molten copper.

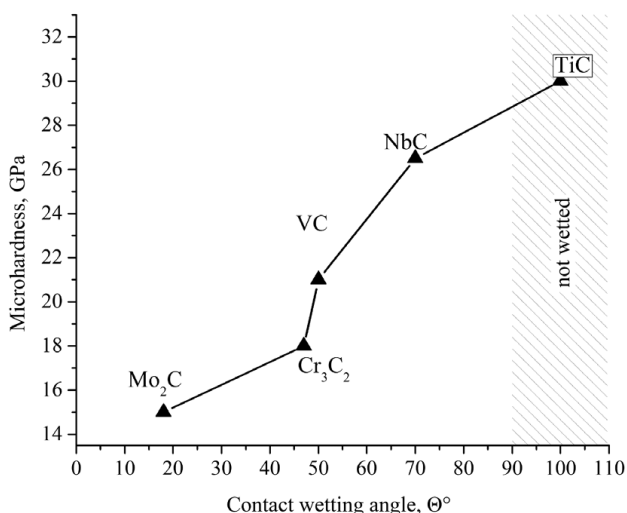


Fig. 1. Comparative characteristics of the wetting capacity of copper and the microhardness of certain refractory carbides of metals from group IV–VI in the periodic system [10]

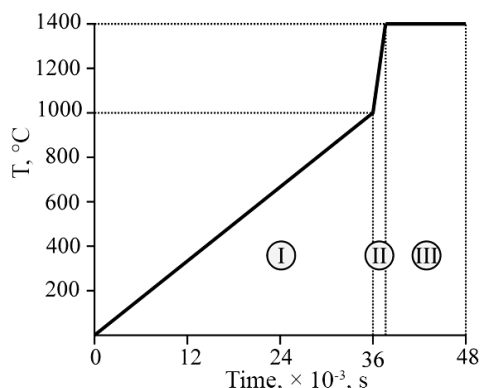


Fig. 2. Mode of workpiece heating in order to obtain NbC–Cu composites

To study the microstructure, we used the scanning microscope Zeiss EVO 40XVP, equipped with the system for X-ray microanalysis INCA Energy and the phase contrast detector CZBSD. In order to determine a phase composition of the obtained materials, we applied the diffractometer DRON-3, under the mode of monochrome CuKα radiation. Results of the X-ray analysis were processed employing the software FullProf.

Thermodynamic calculation of the equilibrium number of phases, as well as concentration, was performed using the software JMatPro.

We determined the character of grain distribution in the carbide phase and a copper bonding applying the method of random secants, the distance between which was 5 μm. For

analysis, we chose an area the size of 0.7×1.0 mm from the photograph of the microsection, acquired from electron microscopy under the mode of diffraction of inversely scattered electrons.

Durometric study was conducted using the microhardness meter PMT-3, equipped with the digital camera-eyepiece eTREK UCMOS 5100 (China). Loading on the indenter varied from 0.5 to 2 N. The microhardness value was calculated based on the results of three imprints from formula:

$$H_{\mu} = 1,854 \frac{F \cdot 10^5}{d^2}, \quad (1)$$

where H_{μ} is the microhardness, MPa; F is the load, N; d is the diagonal of pyramidal imprint, μm.

Macrohardness in line with the Rockwell method was determined according to a standard procedure from GOST 9013-59 (ISO 6508-86) at the hardness meter TK-2 on scale C.

The boundary strength at bending (R_{bm}) was determined by the method of a three-point bending according to GOST 20019-74 on samples the size of 5×5×5 mm; distance between immobile supports was $l=25$ mm. Calculation was carried out according to the following formula:

$$R_{bm} = \frac{3Pl}{(2bh^2)}, \quad (2)$$

where P is the destructive effort, N; b is the width of the sample, mm; h is the height of the sample, mm.

In order to estimate crack resistance, we calculated a critical factor for the intensity of stresses I in the deformation mode (K_{Ic}) using an indirect procedure by Evans-Charles based on the results of measuring the width of cracks that originate from the imprint of the diamond Vickers pyramid. The measurements were carried out at the hardness meter TP-7P-1 under a load of ~600 N; calculation is in line with formula:

$$K_{Ic} = 0,075 \frac{P}{C^2}, \quad (3)$$

where K_{Ic} is the coefficient of crack resistance, MPa·m^{1/2}; P is the load on indenter, N; C is the length of a crack, measured from the middle of the imprint (Fig. 3).

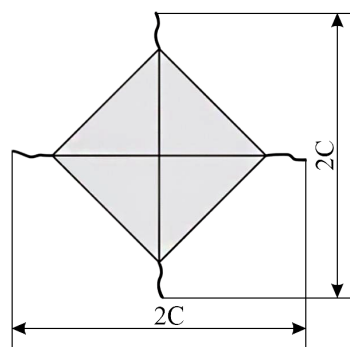


Fig. 3. Schematic of crack length measurements when determining crack resistance

Considering that, according to [12], coatings from the system refractory compound-copper are promising for the

electric-spark application of antifriction coatings, the developed composite materials of the system NbC–Cu were applied onto steel of grade 45 by the electric-spark method. In this case, we used the manual installation MP-EL2, equipped with an electromagnetic vibrator, under the following modes: frequency, 200 Hz; voltage of idling, 39 V, the average magnitude of current in the circuit of capacitors charge, 1.2 A. Duration of treatment changed in the range from 1 to 8 min/cm². We applied the coatings in the open air. The kinetics of coating application (a change in the mass of the anode and cathode) were estimated by weighing the samples after a coating application at a step of 1 min/cm². Based on the acquired data, we built kinetic curves for the total change in the mass of the anode $\Sigma\Delta a$ and the cathode $\Sigma\Delta k$. A mass transfer coefficient was calculated from formula $K=(\Sigma\Delta k/\Sigma\Delta a)\cdot 100\%$. The weighing was performed at the analytical scale VLA-200.

Tribotechnical properties of the applied coatings were determined at the friction machine SMC-2 in line with the scheme disc-pad; the coating was applied to the inner surface of the pad whose counterbody was a roller made of steel 45, thermally treated to a hardness of 40 HRC. In the process of the tribotechnical tests, we measured a friction coefficient and temperature in the area of contact. Friction coefficient was calculated based on the measured friction momentum, which was registered using an analog-to-digital converter. The temperature was determined based on the readings from a thermocouple, placed at a distance of 1 mm from the contact zone. Relative wear resistance was determined in line with the procedure proposed in paper [13]. A given procedure implied the establishment of mass wear in samples with the examined coatings under conditions of their friction against loose abrasive. The reference was a sample with the coating from the solid alloy VK6.

5. Results of studying the structure and properties of the composite Cu-NbC, as well as coatings based on it

The results of microscopic analysis of the starting NbC powder (Fig. 4, *a*) show that it consists of dispersed (the size of ~1 μm) carbide particles with the developed surface. After we impregnated the pressed powder with molten copper, we obtained a composite whose structure consists of the carbide frame, formed from grains of rounded shape and the layers of a metallic bonding (Fig. 4, *b*). The structure of the material forms by the dissolving-deposition mechanism, characteristic of the process of liquid-phase sintering under conditions for good ($\Theta \ll 90^\circ$) wetting of the refractory component with molten metal-bonding. Given that, according to data from paper [14], in the system NbC–Cu at a temperature of 1,150 °C the contact wetting angle Θ is $< 90^\circ$ (at which impregnation is fundamentally impossible), then the progress of the impregnation process at 1,400 °C indicates a strong temperature dependence of the contact wetting angle in the system NbC–Cu. The process of impregnation is accompanied by an increase in the size of grains in the carbide phase, compared to the starting powder, as well as the presence of some grains of pores with a spherical shape in the body. The results of the X-ray phase analysis (Fig. 5) revealed only two phases of NbC with a crystalline parameter of 4.468 Å, which corresponds to the formula of NbC_{0.97} and Cu with a crystalline parameter of 3.625 Å (increased by

0.001 nm). Such a character of change in the phase crystalline parameters is predetermined by the limited mutual solubility between a carbide and a metallic phase, which occurs at impregnation temperature (1,400 °C). The results of analysis into the distribution of carbide grains based on size using the method of random secants (Fig. 6) show that their average size is ~1.8 μm , while the greatest quantity (~40 %) accounts for grains the size from 0.5 to 1.0 μm . The character of distribution of elements at the interphase boundaries was determined by the relative intensity of spectra in the characteristic X-ray radiation from Nb and Cu ($I_{\text{Nb, Cu}}$). Width of the diffusion zone was measured based on zones in which the high intensity of radiation was observed at once for Nb and Cu. Results from the microrentgenospectral analysis along the line that passes through the interphase boundaries (Fig. 7) indicate the presence of a diffusion zone with a thickness of 2–3 μm , formed as a result of redistribution of Nb and Cu. Its presence at the interphase boundary is a positive factor, as it greatly enhances the bond between a metallic and a carbide phase, making it possible to obtain a high level of mechanical properties. Hardness of the resulting material is ~40 HRC, which is 1.2 times higher compared with materials of the system Cu-WC with same volumetric ratio between the carbide and metallic phases; fracture toughness is $\text{MPa}\cdot\text{m}^{1/2}$, strength at bending is 600 MPa.

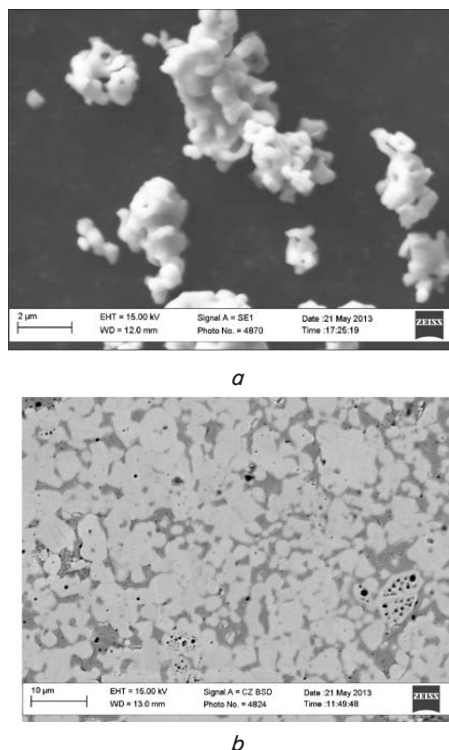


Fig. 4. Results of microscopic analysis: *a* – morphology of particles in the starting NbC powder; *b* – structure of the NbC–Cu composite after impregnation

The results from determining the kinetics of a mass transfer (Fig. 8) in the process of applying a coating from the developed composite by using the method of electric-spark doping demonstrate that the most optimal treatment mode is 3 min/cm². Further increase in the duration of treatment leads to a dramatic decrease in the coefficient of mass transfer.

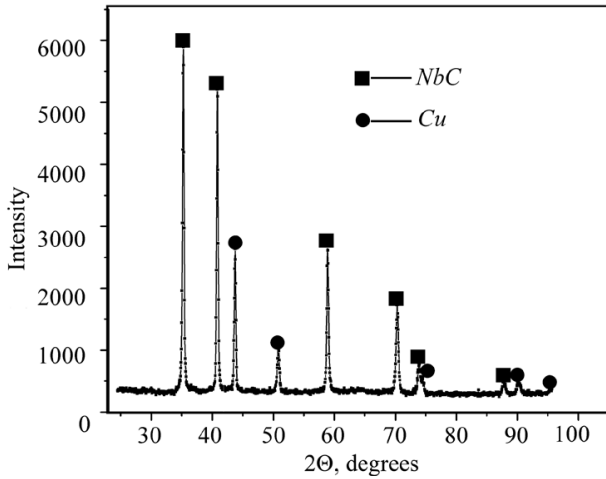


Fig. 5. Diffractogram of the NbC–Cu composite

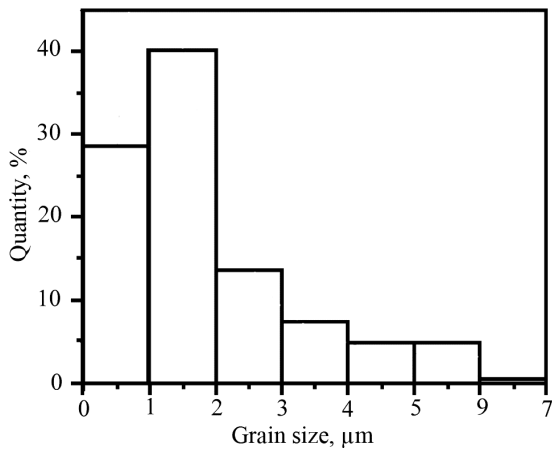


Fig. 6. Analysis of size distribution of NbC grains in the structure of NbC–Cu composite

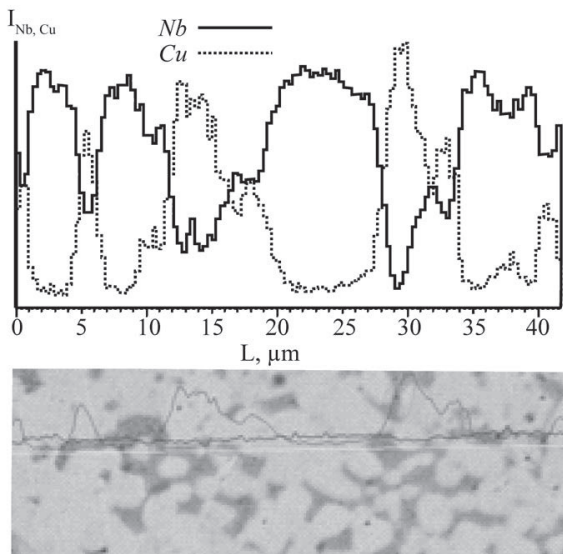


Fig. 7. Distribution of chemical elements at the interphase boundaries in NbC–Cu composite

Microscopic analysis into morphology of the applied coating (Fig. 9) reveals that the surface is relatively smooth while pores and cracks are practically absent. In the surface layer, one observes the disperse, evenly distributed, inclu-

sions of the phase rich in niobium (based on the results of energy-dispersion analysis, the content of Nb is ~69%). The thickness of the applied layer is ~30 μm , its microhardness is ~500 MPa.

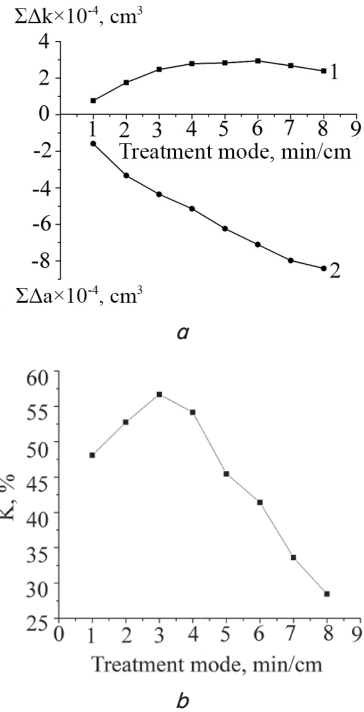


Fig. 8. Dependence of mass transfer of the electrode from NbC–Cu composite (anode) to the steel surface (cathode) depending on the treatment mode: *a* – change in the mass of cathode and anode; *b* – changing in the coefficient of mass transfer

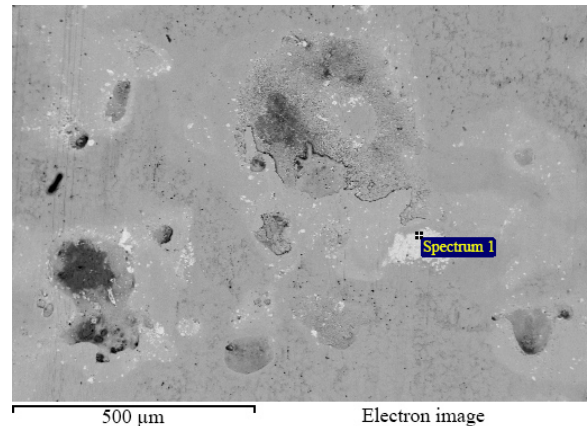


Fig. 9. Morphology of the surface layer from NbC–Cu composite applied using electric-spark doping

Results of the tribotechnical study show that under these testing conditions the values for a friction coefficient at the first stages of testing admit values of ~0.1. Over the first 1,000 s of testing there occurs the intensive growth of a friction coefficient to a value of ~0.16 (Fig. 10). The temperature in this case rapidly rises and accepts a value of ~90 $^\circ\text{C}$. Further increase in the duration of testing leads to a smooth decrease in both the temperature and friction coefficient; in 5,500 s of operation the values for both parameters stabilize at the level $\mu=0.04\text{--}0.05$ and $T=35 \text{ }^\circ\text{C}$.

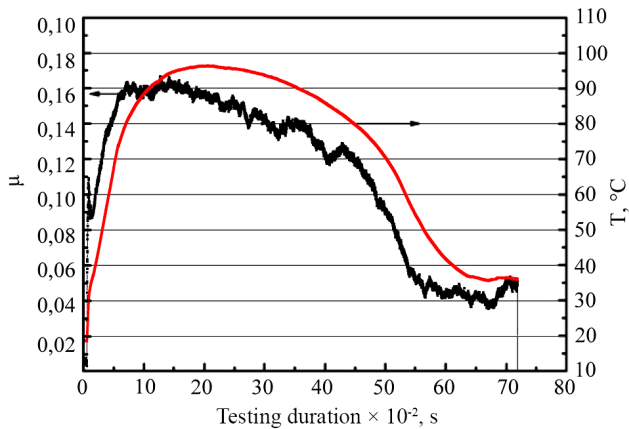


Fig. 10. Kinetics of change in friction coefficient (μ) and temperature (T) depending on the duration of testing for the electric-spark coatings using electrodes from the system NbC–Cu

The results from determining wear resistance of the coating made from NbC–Cu composite under conditions of friction against loose abrasive indicate that its value is only 10 % lower compared with conventional electric-spark coatings from the alloy VK6.

6. Discussion of results of studying the structure and properties of NbC–Cu composite, as well as coatings based on it

Obtaining composite materials in the system NbC–Cu applying a method of impregnation of porous skeletons is possible only under the condition when a contact wetting angle $\Theta \ll 90^\circ$. At copper melting temperature, its value $\Theta \sim 70^\circ$; under such conditions, the rate of impregnation will be low and there is a danger of the formation of non-impregnated zones. An increase in temperature to 1,400 °C leads to a dramatic increase in mutual solubility, the formation of a diffusion zone, and a decrease in the contact wetting angle. This is also confirmed by the thermodynamic calculations of the phase elemental content (Fig. 11). In this case, no formation of new phases is observed in the process of crystallization of an alloy-bonding. The rounded shape of particles in the carbide phase indicates that in a given system copper is not an interphase-active element, that is, its presence in the system does not lead to a decrease in energy at the boundary between a solid and a melt.

An analysis of the size distribution of NbC grains reveals that the specified parameter is described with high precision (corrected determination coefficient $R^2 \sim 0.95$) by the logarithmic normal law in the form:

$$N(x) = d_0 + \frac{A}{wx\sqrt{2\pi}} e^{-\frac{\left[\ln\frac{x}{x_c}\right]^2}{2w^2}}, \quad (4)$$

where w , d_0 and A are the distribution parameters, $N(x)$ is the amount, %; x_c is the average value for the desired magnitude. For a given case, the values for parameters are: $w=0.55$; $d_0=3.02$; $A=79.4$; $x_c=1.3$. That makes it possible to calculate mechanical properties applying the ratio by Hall-Petch based on the mean value for the size of grains.

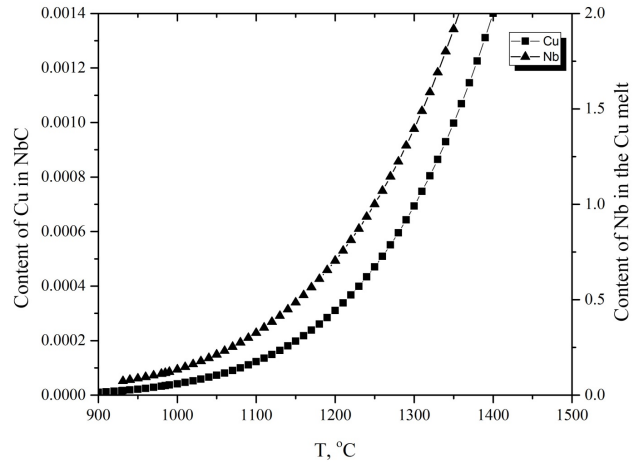


Fig. 11. Temperature dependence of solubility of Cu and Nb in NbC, and in melt, respectively

The use of composites Nb–Cu as the electrode materials for applying coatings via a method of electric-spark doping enables the formation of wear-resistant layers with a high level of anti-friction properties due to the presence of the matrix-reinforced structure in a surface layer. Mass transfer when applying coatings is described by the curve with a maximum corresponding to the mass transfer coefficient equal to 57 %.

The results obtained are justified by the rational choice of the ceramic component of a composite (NbC) and performing the process at a temperature that makes it possible to receive a pore-free material by providing for wetting capacity. The advantage of the developed composite over those known from [3–7, 11] is the simplicity of the technology for obtaining them and a possibility to apply composites in the form of coatings.

The main shortcoming of the electric-spark application of coatings from the NbC–Cu composites is the limit on the thickness of a coating. The maximum possible thickness is 60 μm . In addition, the developed coatings are characterized by the high surface roughness. In order to eliminate it, it is necessary to foresee a period for the alignment of a friction pair. Advancement of this research implies the employment of additional operations to reduce a coating’s roughness, for example by the method of rolling. It is also promising to apply combined coatings from the developed composite and conventional materials for electric-spark doping. However, there may arise difficulties related to the choice of materials and selection of the optimal duration for the process of applying a coating due to the difference in the physical properties of materials.

7. Conclusions

1. It was established that the optimal temperature for obtaining the NbC–Cu composites using the method of impregnation of pre-formed carbide skeletons is 1,400 °C; under these conditions, there is a significant decrease in the contact wetting angle with a respective increase in the rate of impregnation.

2. It was established that the structure of the NbC–Cu composite consists of rounded NbC grains the average size of 1.8 μm , distributed according to the logarithmic-normal law and the copper matrix. Such a shape of grains indicates structure formation in line with the mechanism of deposition dissolution, characteristic of solid metallic-ceramic alloys.

3. We have selected the following optimal technological regimes for applying the coatings from Cu-NbC composites:

treatment duration is 3 min/cm²; current is 1.2 A, voltage is 39 V, vibration frequency is 200 Hz.

4. It was established that the coatings from NbC–Cu composites consist of a matrix, which is an alloy of copper and iron, the structure of which contains the evenly dis-

persed particles of NbC. Coating thickness is 30 μm; its microhardness is ~500 MPa. Under conditions of friction without lubrication against a steel counterbody, the friction coefficient after alignment is 0.04. That makes it possible to use the developed coatings as the antifriction ones.

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