

Original Article

Increase in properties of copper electrical contacts in formation of composite coatings based on Ni-C-Ag-N system



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ABSTRACT

The article is concerned with analyzing the structure and properties of the Ni–C–Ag–N coating formed on copper by the complex method combining the electroexplosive spraying of coating, its irradiation by a pulsed electron beam and a subsequent nitriding in plasma of a gas discharge of low pressure. The structural constituents of coating's surface after the electroexplosive spraying as well as after the electron-beam processing and nitriding are studied. It is shown that a coating's thickness amounts to $\approx 90 \ \mu\text{m}$. The wear resistance of a copper sample with the deposited coating exceeds that of the copper without the coating by ≈ 1.6 times. The friction coefficient of samples with the coating ($\mu = 0.5$) is less than that of the copper without the coating ($\mu = 0.679$) by ≈ 1.35 times. It is established that the hardness of the coating increases as the substrate is approached and reaches the maximum value of ≈ 1780 MPa (the hardness of substrate is 1300 MPa). By means of micro-X-ray-spectral analysis it is detected that the main chemical element of the coating is silver, but copper, nickel, carbon and nitrogen are present in a considerably smaller quantity. It is established by the methods of diffraction electron microscopy that the main phases of the coating are solid solutions.

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1. Introduction

Nowadays, none of appliances working with the use of electric current [1] can exist and function without electrical contacts. In a general case, an electrical contact consists of two contact parts being items of separate supply and installed on different parts of a product [2]. That is why along with the requirements of ensuring electrical and mechanical connections [3] the contacts should meet such a requirement as interchangeability [4]. It means that when connecting any of several standard elements of replacement to a base unit the parameters of the equipment should be invariable. Furthermore, all main characteristics of an electrical contact should be stable in time and in the process of action of any external factors [5] as well. The main factors are the vibration, mechanical shocks, acoustic noise, increased and reduced pressure, humidity and temperature of environment, atmospheric precipitations, static and dynamic dust, salt fog and other corrosive agents of environment [6].

In order to reduce the overall dimensions of the equipment in conditions of extremely complicated circuitries, the electric switch should ensure a presence of maximum quantity of electric circuits being switched on and switched off simultaneously [7]. The requirement suggests the presence of a large number of contacts (contact pairs) in one switch, and currents from 10^{-6} to 50 A at working voltages from 10^{-3} to 10^{3} V [8] can pass through them. The parameters refer to common switches, and special switches may have other characteristics as well. Working frequencies of electric current passing through switches may reach 100 Hz. The enumerated requirements characterize the working conditions of electric switches and their technical characteristics only in general degree but they estimate correctly enough the complexity of the problems that the developers of switches have to solve with the aim of providing the high quality of them. According to the estimates of specialists 50% of failures occur due to the insufficient quality of electric switches [9]. That is why a worldwide struggle for quality of electric switches is going on in conditions of expansion the functions and complication of equipment.

The main tendencies for increasing the quality of connectors are rather evident from the technical point of view. To these belong the development of new original constructive solutions, the selection and development of materials of higher quality for fabricating the switches, the development of new technological processes for manufacturing the switches, the provision for reducing the effect of external factors directly on electric switch, the development of new methods of switch mounting in equipment [10]. Activities in all directions occur, as a rule, in combination with a simultaneous achievement of a predetermined purpose, resulting in a substantial increase in quality and a simultaneous decrease in cost of switches [11].

Nowadays the investigations directed to creation of different high-voltage switches of direct and alternating current [12,13] are developed intensively. Majority of versions of such switches are developed. Some of them contain complicated circuit solutions based on use of thyristors [14], semi-stors [15] and others. Depending on design, such appliances have a number of disadvantages: the difficulty in switching off [16], complexity of circuit [17], high cost. For these reasons the reliable and simple in design switch apparatuses [18,19] are used for commutation of powerful electrical networks. As a rule, the basis for such apparatuses is the process of closing and opening the paired contacts [20]. The increase in on/off cycles of a commutation switching apparatus [21] results in a gradual failure of an electrical contact surface [22].

One of the tasks of modern materials science is a search for ways to increase the service life of different components through formation of the best structure [23]. It is also possible to ensure a minimum economical cost of components by means of materials science. Applying the tasks to electrical contacts of switches of powerful electric networks one may speak of a cost minimization for components with a high electrical conductivity based on silver, gold, etc. It is possible to decrease in costs and increase in a service life of electrical contacts by application of economical substrates, for example those, made of copper, aluminum and other materials on the surface of which the highly efficient electroerosion-resistant coatings are deposited.

Electrical contacts based on the Ni–C–Ag system are intended for closing and opening the electric circuits in apparatuses with a voltage of up to 1000 V [24]. The principal method of their manufacturing is the method of powder metallurgy using silver-containing compositions with a highly dispersed structure. Electrical contacts based on the Ni–C–Ag system are used in automatic switches, magnet starters, contactors, controllers and relays of different grades [25]. The resistivity of the contacts depending on their grades vary from 0.019 to 0.07 mOhm/m [26]. Contacts of a contactor, flexible connections are given spare parts are replaced without special adjustment and without use of special tool [27]. For majority of

Table 1 – Processing modes of electroerosive tantalum coatings by low-energy high-current electron beam and subsequent nitriding.					
Mode of complex processing	Parameters of low-e	energy high-current el	Parameters of nitriding		
	Surface density of energy (E _s), J/cM ²	Duration of pulses (t), μs	Number of pulses (N)		
1	20	200	5	Time (τ), 5 h; temperature (T), 520 $^\circ$ C	
2	30				
3	40				
4	20		3	Time ($ au$), 3 h, temperature (T), 600 °C	
5	30				
6	40				
7	40	150			



Fig. 1 – Photographs of the samples' surface after electroerosive spraying of the Ni-C-Ag-system coatings.

contactors both movable and stationary contacts, that are placed in contact unit of product, are used [28]. The amount of the required contacts is different for different types of contactors and depends on various factors [29]. In view of the fact that electrical contacts of contractors are produced at plants worldwide their size may be slightly different [30]. So far the application of the Ni–C–Ag–N system has not been realized. However, the high-strength carbo-nitrides and nitrides of metals will result in an increase if the wear resistance, hardness and electroerosion resistance of electrical contacts. It



emphasizes the persistence and advanced character of the research and its scientific novelty.

A selection of the method of electroexplosive spraying for a formation of coating in the research is due to perspectives of its development and advantages. The electroexplosion spraying is actively studied not only the team of scientists who had written the given paper but also, for example, by Chinese scientists [31]. The electroexplosion spraying is performed in order to give necessary properties to surfaces of metallic and nonmetallic products. These may be an increase in the electric and heat conductivity, strength, protection from the effect of corrosion processes, recovery of geometrical size, etc. In this case, the necessary equipment, expendable materials and technology of execution of spraying are selected in relation to a specific problem depending on a product's material. Metallic powder or mixtures, in whose composition a ceramic powder in definite amounts is introduced in addition to metallic component, may be sprayed. It is also possible to use different foils. It cheapens greatly the technology of formation coating and has no effect on their properties. The main advantages of the electroexplosion spraying are the following:

- It is possible to work in any climatic conditions (pressure, temperature, humidity);
- It is possible to use equipment of a stationary and movable type, which, in the latter case, allows works to be undertaken in place of work execution;

- It possible to apply coatings to local portions (defective sites);
- 4. It is possible to form layers with different properties;
- It is possible to form a layer of a required thickness or layers with different thickness in multi-layer coatings;
- A process of spraying has no effect on a structure of the product on which the coating is deposited;
- 7. Safety;
- 8. Ecological safety.

In connection with the facts stated above the paper is concerned with forming and analyzing the structure and properties of the Ni–C–Ag–N coating formed on copper by a complex method combining the electroexplosing spraying, the irradiation by pulsed electron beam and subsequent nitriding in plasma of a gas discharge of low pressure.

2. Materials and methods

The coatings were formed on samples of M00 copper (the chemical composition, mass%: 99.99 Cu, 0.001 Fe, 0.001 Ni, 0.001 S, 0.01 P, 0.001 As, 0.001 Pb, 0.001 Zn, 0.001 Ag, 0.0005 O, 0.0005 Sb, 0.0005 Bi, 0.0005 Sn). The samples were parallelepipeds with sizes $20 \times 20 \times 5$ mm. The electroexplosive spraying of the Ni–C–Ag coating was performed on a setup EVU 60/10M. The process was done at the following parameters: the time of plasma effect on a sample surface ~100



—– portions of coppery colour; —– zones of black colour;

-- areas of grey colour ; -- regions with a noticeable silvery relief

a - a surface of irradiated sample 1;

b– a surface of irradiated sample 2;

c – a surface of irradiated sample 3;

d – a surface of irradiated sample 4;

e - a surface of irradiated sample 5;

f-a surface of irradiated sample 6;

g – a surface of irradiated sample 7

Fig. 3 – Photographs of sample's surface with an electroerosive coating. of the Ni–C–Ag- system after nitriding and pulsedperiodic electron beam processing.

 μ s, the absorbed power density on jet axis ~5.5 GW/m², the pressure in shock-compressed layer near a surface being irradiated ~12.5 MPa, residual gas pressure in a working

chamber ~100 Pa, the plasma temperature on nozzle cut ~ 10^4 K. A three-layer composite electroexplosive material was used for spraying. One of foil layers was made of silver



Fig. 4 – A relation of samples' surface elements with an electroerosive coating of the Ni–C–Ag-system after nitriding and pulsed-periodic electron beam processing.

(the chemical composition, mass%: 99.9 Ag, 0.003 Pb, 0.035 Fe, 0.002 Sb, 0.002 Bi, 0.058 Cu) 640 mg by mass; another one is of nickel (the chemical composition, mass%: 99.99 Ni, 0.005 C, 0.001 Mg, 0.001 Al, 0.001 Si, 0.001 P, 0.001 S, 0.001 Mn, 0.002 Fe, 0.001 Cu, 0.0005 Zn, 0.0005 As, 0.003 Cd, 0.0003 Sn,

0.0003 Pb, 0.003 Bi) 420 mg by mass. Between silver and nickel foil the layer of carbon-graphite fiber 50 mg by mass was placed. After electroerosion spraying the irradiation of the coatings by pulsed electron beam and nitriding were carried out on setup 'COMPLEKS' [32]. The combination of

Table 2 – X-ray phase analysis data of samples with the Ni–C–Ag–N coatings.						
Sample	Phases	Phase content,	CSR, nm	Crystal lattice	Crystal lattice parameter, Å	
	revealed	mass%		A	С	
1	Ag	56.29	83.54	4.0787	_	
	Cu	3.19	21.97	3.619	-	
	NiC _{0.33}	14.86	111.89	2.6776	4.3497	
	Cu _{0.08} Ni _{0.92}	25.65	-	3.5278	-	
2	Ag	50	97.35	4.0818	-	
	Cu	12.72	14.31	3.5606	-	
	NiC _{0.33}	12.96	31.86	2.6699	4.3992	
	Cu _{0.08} Ni _{0.92}	24.32	-	3.532	-	
3	Ag	56.05	270.21	4.0752	-	
	Cu _{0.08} Ni _{0.92}	38.03	8.27	3.5438	-	
	NiC	5.92	-	2.6513	4.434	
4	Ag	69.09	217.4	4.0775	-	
	Cu _{0.08} Ni _{0.92}	6.22	81.25	3.5413	-	
	NiC	24.7	8.28	2.6563	4.4198	
5	Ag	49.65	112.75	4.0803	-	
	Cu _{0.08} Ni _{0.92}	50.23	18.27	3.5307	-	
	NiC	0.12	69.31	2.5971	4.2342	
6	Ag	80.11	49.97	4.0811	-	
	Cu _{0.08} Ni _{0.92}	19.31	29.93	3.5377	-	



Fig. 5 – X-ray diffraction pattern portions of the Ni–C–Ag–N coating.

parameters of electron beam processing and nitriding are listed in Table 1.

The investigation into defect substructure and elemental composition was performed by the methods of scanning electron microscopy (a device Carl Zeiss EV050 with an attachment for energy dispersion X-ray spectral analysis EDS X-Act). The research into the elemental and phase composition, defect substructure state were done on transmission electron diffraction microscope (JEM 2100F, JEOL). The coatings' hardness was measured by means of ultramicrotester



Fig. 6 – The parameters of the phases being formed in the Ni–C–Ag–N coating obtained by the complex method.

Shimadzu DUH-211 ($P_n = 30$ mN). The tribological properties of the coating were studied on a tribometer Pin on Disc and Oscillating TRIBO tester ('TRIBO technique', France) in conditions of dry friction. The test for wear resistance was performed in ball-disc geometry with the following parameters: the SHKH 15 steel ball 6 mm in diameter, loading - 3 N, distance 300 m, wear track radius - 2 mm, velocity of ball travel 25 mm/s. The wear resistance of samples with the coating was calculated after a wear track profilometry. Electromagnetic starters of CJ 20 series with copper contacts, on the surface of which the coating under study was deposited, were tested for the commutation wear resistance with determination of on/ off cycle number. According to AC-3 starters' (direct action) application the tests were done in three-phase circuit of alternating current with inductive loading under the following parameters: the rated voltage 400/230 V, current frequency 50 Hz, current intensity up to 320 A, $\cos \varphi = 0.35$. The electrical conductivity was measured on the same test bench where commutation resistance tests were done.

3. Results and discussion

3.1. Quality control of coatings surface

3.1.1. Relief of coatings' surface after electroexplosive spraying

As a result of electroexplosive spraying of the Ni–C–Ag-system coatings a dense coating of silvery-grey colour with the relief noticeable with the naked eye is formed on the surface of copper substrates. The relief is formed by flow lines, small point silvery inclusions as well as regions with coarse elements of relief.

A group of three regions is located on the surface of sample 1 (Fig. 1, a). The total fraction of regions with coarse elements of relief is the least and amounts to 2.00%. Around them and on a small area in the corner of the sample there are zones of black hues which occupy 24.69% of the coating. The remainder of the sample is presented by the areas of grey-silvery colour on which the traces of surface flows and small lustrous inclusions are observed.

On the surface of sample 2 (Fig. 1, b) the regions with coarse elements of relief are expressed implicitly and are minimal. The total fraction of the regions decreased to its minimum of 1.36%. At the same time the zones of black hues adjacent to them and having irregular shape occupy a smaller area amounting to 24.64%. The remainder of the surface is presented by the areas of grey-silvery colour joined to each other that amount to 74%.

The least surface fraction of sample 3 (Fig. 1, c) is occupied by regions with coarse elements of relief. Their area reached its maximum of 12.4%. The zones of black hues whose surface fraction increased to 34.4% extend from a large region with the relief to the opposite corner along the diagonal. The largest part of the coating is occupied by the areas of silvery-grey hues. Their surface fraction decreased to 53.51%.

In the coating deposited to sample 4 (Figure, d) the large silvery portions are observed for the first time, and their total area is 6.28% of the surface and is minimal. The fraction of the coating occupied by coarse elements of relief decreased to



Fig. 7 – A characteristic dependence of the contact resistance (R) on number of on/off cycles (N) in tests of electroerosive coatings of the N–Ni–C–Ag system for. the electroerosion resistance in conditions of arc erosion.



Fig. 8 – A characteristic time-dependence of contact voltage of paired contacts A (black curve) and B (red curve) with closing of one phase. b - a magnified image of the portion indicated by arrow on (a).

9.68%. The zones of black hue reach the minimum and amount to 16.96%. The areas of grey-silvery colour occupy the largest part of the coating. Their surface fraction increased to 67.08%.

In sample 5 (Fig. 1, e) the least fraction of surface whose value decreased to 2.92% is occupied by the regions with coarse elements of relief. The regions are grouped mainly nearer to samples' boundaries. The fraction of the coating occupied by zones of black hues reaches its maximum of 47.24%. The areas of grey-silvery colour occupy two remaining portions. Their total area decreased to 49.84%.

In sample 6 (Fig. 1,f) the regions with coarse elements of relief occupy the smallest area. Their surface fraction increased to 8.37%. The zones of black hues locate around the

coating under study and in one of samples' corners. Their total surface fraction decreased to 22.48%. The areas of grey-silvery colour whose total area increased to 69.15% occupy the remainder of the coating.

In sample 7 (Fig. 1, g) the regions with coarse elements of relief are grouped mainly in the central part of the sample, and their minimal surface fraction decreased to 3.06%. On the obtained coating, along one of its boundaries there is a group of silvery portions whose total surface fraction decreased to 4.83%. The zones of black hues, located at samples boundaries and encircling the region with coarse elements of relief in its central part, occupy totally the surface fraction whose value increased to 39.21%. The remainder (52.90%) of the coating is presented by the areas of grey-silvery colour.



Duration of tests, s

Fig. 9 – The friction coefficient as a function of duration of sample's tribological tests. a – copper with the Ni–C–Ag–N coating, b – copper without the coating.

For a more visual presentation of relation of sample surface elements with electroexplosive coating of the Ni–C–Ag system the bar graph (Fig. 2) was constructed (Fig. 2).

3.1.2. Surface relief of electroexplosive coatings after nitriding and electron beam processing

After nitriding and pulsed-periodic electron beam processing of the Ni–C–Ag coating some changes are observed in it. The samples' surface decolorizes and pores are formed in it. In some places the coating is absent and, instead of it, the areas of coppery colour are observed.

On the surface of sample 1, along its boundaries the regions with noticeable silvery relief are observed, their fraction



Fig. 10 – Hardness of the Ni–C–Ag–N/substrate system as a function of a coating surface. Hardness of copper without the coating is 1300 MPa. Dotted lines indicate the Ni–C–Ag–N coating/substrate transition layer.

is minimal and amounts to 0.80%. The portions of coppery colour with area of 4.52% locate at its edges as well. The remainder of the coating is presented by the area of grey colour, on which pores and compactions along boundaries with other regions, are seen. The surface fraction of the zone under consideration is the greatest and amounts to 94.68%.

The fraction of the coating deposited on sample 2 (Fig. 3,b) belonging to the zone of black colour is equal to 5.16%. The area occupied by portions of coppery colour increased to 26.60%. The area of grey colour occupies the largest part of the coating. Its fraction decreased to 68.24%.

On the surface of sample 3 (Fig. 3, c) the regions with noticeable silvery relief appear again, in this case their total area amounts to 1.26%. The fraction of the coating belonging to two zones of black colour increased to 6.87%. The portions of coppery colour increased their presence to the maximum of 31.74%. The remainder of the coating is presented by the areas of grey colour whose surface fraction decreased to 60.13%.

The surface fraction of sample 4 (Fig. 3, d) occupied by portion of coppery colour decreased to 2.92%. The areas of grey colour having curved boundaries decreased their presence to 32.15%. The zones of black colour whose surface fraction increased to 64.92% occupy the largest part of the coating.

On the surface of sample 5 (Fig. 3, e) the small portions of coppery colour locate chaotically, their area reached its minimum of 1.27%. The regions with noticeable silvery relief that are nonuniformly scattered in the coating occupy the fraction of 3.13%. The remainder of the coating is presented by the zones of black colour whose surface fraction reached its maximum of 95.60%.

The fraction of the coating deposited to sample 6 (Fig. 3,f) and belonging to the zone of black colour decreased to 3.40%. The area occupied by the portions of coppery colour increased to 6.82%. The fraction of the coating that reached its maximum of 3.13% belongs to the regions with noticeable



Fig. 11 — A structure of the Ni–C–Ag–N coating formed on copper. Scanning electron microscopy. Transverse metallographic section. The coating is indicated by the arrow.

silvery relief. The area of grey colour that increased its presence to 86.41% occupies the largest part of the coating.

The fraction of the coating deposited on sample 7 (Fig. 3, g) belonging to the regions with a noticeable silvery relief decreased to 2.42%. The surface with portions of coppery colour increased to 10.46%. The largest fraction of the coating belongs to the areas of grey colour amounting to 87.12%.

For a more visual presentation of relation of samples' surface elements with the electroerosive coating of the Ni-C-Ag-system after nitriding and electron beam processing the bar graph (Fig. 4) was built.

3.2. Research into the coatings by the method of X-ray phase analysis

The data of X-ray phase analysis on the samples with the Ni–Ag–N-coatings are listed in Table 2. Let us analyze the data.

The four phases were revealed in the sample irradiated by mode1 (Fig. 5, a) by means of X-ray phase analysis. The Agphase possesses the largest mass fraction, its content amounts to 56.29%. The parameter of crystal lattice a in the Agphase amounts to 4.0787 Å, in the coherent scattering



Fig. 12 – A distribution of chemical elements in the Ni–C–Ag–N coating formed by a complex method. Scanning electron microscopy, mapping method. a – an image formed by a superposition of images obtained in characteristic X-ray radiation of Cu (b), Ag (c), Ni (d) atoms.



Fig. 13 – A distribution of chemical elements in the Ni–C–Ag–N coating formed by a complex method. STEM, mapping method. a – a bright-field image, b–d – images obtained in characteristic X-ray radiations of Ag, Cu and Ni atoms. 1 – results of mapping of a coating's surface layer. 2 – results of mapping of the layer at a boundary with a substrate.

Table 3 – Elemental composition of the coatings' surface layer.					
Element	E, keV	Mass. %	Quantity	Error, %	At. %
Ni (K)	7.471	1.13	10098	0.17	1.83
Cu (K)	8.040	17.08	135198	0.01	25.70
Ag (L)	2.984	81.79	352046	0.00	72.47
Total	-	100	—	-	100

region (CSR) it is 83.54 nm. At the same time the content of the $Cu_{0.08}Ni_{0.92}$ phase amounts to 26.65%. In this case, the parameter of its crystal lattice a is 3.5278 Å. Mass fraction of the NiC_{0.33} phase is equal to 14.86%, whose crystal lattice parameters a and c have values of 2.6776 Å and 4.3497 Å, respectively, and the CSR is 111.89 nm. The Cu-phase possesses the least mass fraction of 3.19%. In this case, its parameter of crystal lattice a and the CSR have values of 3.619 Å and 21.97 nm.

In the sample irradiated by mode 2 (Fig. 5, b) four phases were detected. The Ag-phase has the largest mass fraction, its content decreased to 50%. The crystal lattice parameter a increases to 4.0818 Å, at the same time the CSR of the phase under consideration increased to 97.35 nm. The content of the $Cu_{0.08}Ni_{0.92}$ phase decreases to 24.32%. The crystal lattice parameter a is equal to 3.532 Å. The mass fraction of the $NiC_{0.33}$ phase decreased to 12.96%, in this case the crystal lattice parameter c increased to its maximum of 4.3992 Å. The crystal lattice parameter a decreased to 2.6699 Å. The CSR of the phase under consideration decreased to 31.86%. The Cu-phase in the sample under study possesses the minimum mass fraction, its content reached its maximum of 12.72%. In this case the CSR and crystal lattice parameter a decreased to 14.31 Å and 3.5606 nm.

In the sample irradiated by mode 3 (Fig. 5, c) three phases were revealed among which the Ag-phase possesses the largest mass fraction. The Ag content increased to 56.05%. In this case the crystal lattice parameter a reached its minimum of 4.0752 Å. The CSR value, on the contrary, increased to its maximum value of 270.21 nm. The mass fraction of the $Cu_{o.08}Ni_{0.92}$ -phase increased to 38.03%. The crystal lattice parameter a of the $Cu_{o.08}Ni_{0.92}$ - phase increases to its maximum value of 3.5438 Å. The CSR value amounts to 8.27 nm. The NiC-phase has the least mass fraction of 5.92% in the sample under study. The crystal lattice parameter a is equal to 2.6513 Å. At the same time the parameter c has the maximum value of 4.434 Å.

In the sample irradiated by mode 4 (Fig. 5, d) three phases were detected, among which the Ag-phase possesses the largest mass fraction, its content increased to 69.09%. In this case the crystal lattice parameter a increased to 4.0775 Å but

Table 4 — Elemental composition of the layer at a boundary with a substrate.						
Element	E, keV	Mass. %	Quantity	Error, %	At. %	
Ni (K)	7.471	0.53	4320	0.33	0.67	
Cu (K)	8.040	60.93	445690	0.00	72.36	
Ag (L)	2.984	38.54	153338	0.01	26.97	
Total	-	100	_	-	100	

the CSR value, on the contrary, decreased to 217.4 nm. The mass fraction of the NiC-phase reaches its maximum of 24.7%. The crystal lattice parameter a increases to 2.6563 Å, at the same time the parameter c decreases to 4.4198 Å. The CSR value is equal to 8.28 nm. The $Cu_{0.08}Ni_{0.92}$ -phase possesses the least mass fraction in the sample under study, its value decreased to its minimum of 6.22%. The crystal lattice parameter a decreased to 3.5413 Å, and the CSR of the phase under study reaches its maximum of 81.25 nm.

In the sample irradiated by mode 5 (Fig. 5, e) three phases were detected, the Cu_{0.08}Ni_{0.92}-phase possessing the largest mass fraction among them. Its content reached its maximum of 50.23%.The crystal lattice parameter a and the CSR decreased to 3.5207 Å and 18.27 nm. The Ag-phase mass fraction decreased to its minimum of 49.65%. The crystal lattice parameter a increased to 4.0803 Å. In this case the CSR value decreased to 112.75 nm. The NiC-phase possesses the least mass fraction in the sample under study, its content decreased to 0.12%. The crystal lattice parameters a and c decreased to 2.597 Å and 4.2342 Å respectively. The CSR value reached its maximum of 69.31 nm.

Three phases were revealed in the sample irradiated by mode 6 (Fig. 5,f), the Ag-phase possessing the largest mass fraction among them. Its content reached its maximum of 80.11%. The crystal lattice parameter a increased to 4.0811 Å, and the CSR value decreased to its minimum of 49.97 nm. The $Cu_{0.08}Ni_{0.92}$ - phase mass fraction increased to 19.31%. In this case, the crystal lattice parameter a decreased to 3.5377 Å. The NiC-phase possesses the least mass fraction in the sample under study, its content increasing to 0.58%. The crystal lattice parameters a and c increased to 2.6694 Å and 4.3462 Å respectively, but the CSR decreased to 19.65 nm.

In the sample irradiated by mode 7 (Fig. 5, g) three phases were identified, the Ag-phase possessing the largest mass fraction among them. Its content decreased to 64.43%. The crystal lattice parameter a reached its maximum of 4.0828 Å, and the CSR value increased to 68.47 nm. The Cu_{0.08}Ni_{0.92}phase mass fraction decreased to 19.31%. In this case, the crystal lattice parameter a and the CSR increased to 3.5377 Å and 29.93 nm. The NiC —phase possesses the least mass fraction in the sample under study. Its content increased to 13.39%. The crystal lattice parameter a reached its maximum of 2.6695 Å. The crystal lattice parameter c increased to 4.3581 Å. The CSR of the phase under study increased to 43.05 nm.

The changes in the crystal lattice parameters of phases forming them and their coherent-scattering regions, as well as the phase content described above are presented as a function of change in irradiation mode in Fig. 6.

3.3. Testing for commutation wear resistance

In the present research, all seven variants with Ni–C–Ag–N coatings formed in all modes of irradiation were tested for the commutation wear resistance. But the best characteristics among all samples were shown by the coating obtained by mode4 (parameters of electron beam processing: 17 keV, 20 J/ cm², 200 s, 5 pul, 0.3 s^{-1} ; parameters of nitriding: 600 °C, 3 h). Further, the description of characteristics under study precisely for this irradiation mode in testing's for commutation wear resistance is given.



Fig. 14 - (a) - a copper sample portion with the coating; (b), distribution of chemical elements in thickness of the sample.

As a result of tests for commutation wear resistance it is established that the coating formed withstands 7005 on/off cycles (Fig. 7). The resistance of the paired electrical contact is less than 15 Ohm in all three phases L1–L3 in the process of testings. It can be noted that the coating obtained by the combined method increases the commutation wear resistance of the ZnO–Ag-system electroerosive coatings by 1000 on/off cycles [33]. The electrical resistance of the ZnO–Agsystem coatings is less than 15 μ Ohm as well. The electrical conductivity of the coatings formed is close to that of silver [33] and amounts to 62.0 MS/m.

At the start of tests the values of electrical resistance are minimal for phases L1, L2 and L3 and amount to 5.3, 4.9 and 4.2 μ Ohm (Fig. 7) with the number of on/off cycles of 201, 172 and 114 respectively. Later on the function of electrical resistance for phase L2 increases continuously to its maximum of 13.5



Fig. 15 – Results of energy spectra analysis of chemical elements being present in coating (averaging was done using three tracks located in parallel); X – a distance from a surface of the coating.

 μ Ohm with 3.990 on/off cycles. At the same time the resistance of phase L1 increases to 8.8 µOhm with 973 on/off cycles, after which the decrease to 8.7 μ Ohm with 2025 on/off cycles is observed and is followed by monotonic growth to maximum value of 13.9 µOhm with 3952 on/off cycles. The parameter under consideration increases in phase L3 to 7.5 µOhm with 1161 on/off cycles, after which its value remains constant up to 1893 on/off cycles. Later on, similarly to phases L1, L2, the resistance of L3 reaches its maximum of 12.9 µOhm with 3973 on/off cycles, and monotonic decrease in resistance is observed to the end of the test for the electroerosion resistance. For phase L1 the value of electroerosion resistance decreases to 8.0 µOhm with on/off cycles increased to 7050. The resistance of phase L2 decreases to 7.4 µOhm with on/off cycles being equal to 7124. For phase L3 the resistance reaches 6.9 μOhm with 7005 on/off cycles.

The dependences of contact voltage of paired contacts were determined in a single-phase closing, single phase breaking and zero current as well as in single-phase breaking and maximum current. As an example the dependence of contact voltage of paired contacts in closing of one phase is given in Fig. 8. From Fig. 8 it is seen that time necessary for contacts to turn off is ≈ 25 s. With breaking of one phase and zero current as well as with breaking of one phase and maximum current the time for contacts to turn off is 20–25 µsecs. The given values of turn off time correspond to modern commutation apparatuses being offered in the market. The Ni–C–Ag –N-system coating formed provides a fast arc quenching in disconnecting the electrical contacts. This opens a prospect of applying such contacts in fast-responding commutation apparatuses.

Since only the coating of Ni–C–Ag–N-system obtained in mode 4 is characterized by the most uniform surface after formation, the silver-base phase composition, NiC and $Cu_{0.8}Ni_{0.92}$, also demonstrates the best electroerosion

resistance in arc erosion conditions, the investigations described below were carried out only for mode 4 of coating formation.

3.4. Final research into structure and properties

As a result of tribological tests it is stated that the wear parameter (the quantity reciprocal to wear resistance) of a copper sample with the Ni-C-Ag-N coating amounts to $5.9 \cdot 10^{-5}$ mm³/Nm and is 1.6 times higher than the wear resistance of copper without the coating (9.6•10⁻⁵mm³/Nm). A friction pair of contacting materials in a geometry "ball" (Shkh 15 steel) - disc (the Ni-C-Ag-N system coating) show their joint characteristic independent of contact area of bodies the friction coefficient. In the present research a comparison of the given characteristic was made with the similar one, but in geometry' ball (ShKh 15 stell) - disc (copper of M00 grade). An absence of a liquid or gaseous interlayer between contacting bodies in friction corresponds to working conditions of the contact pair, commutating the powerful electric networks. Under the mechanical action of the contact pair of electrical contact to each other, only displacement of separate crystals or other structural units relative to each other will be observed, and a mechanism of the failure process will be reduced to merely the phenomena of dry friction along boundaries of structural formations. The friction coefficient of samples with the coating ($\mu = 0.503$) is 1.35 times smaller than that of the copper without the coating ($\mu = 0.679$). If we compare these results with the data published earlier [33], where the Ni-Ag-N-system coating were formed, the following facts can be noted. In the paper [33] the wear resistance of a copper sample with the deposited Ni-Ag-N coating is 13% higher than that without the coating. In the paper [34] the same mode parameters as in the present research were used. The difference is only in the introduction of a carbon-graphite fiber into a composition of a conductor being exploded. The introduction of a carbon-graphite fiber enables the higher wear resistance and lower friction coefficient to be reached.

The friction coefficient under tribological tests of copper and copper with Ni–C–Ag–N- coating varies in different way. The data presented in Fig. 9 testify that the sample with the coating is characterized by a longer stage of running – in, as compared to the sample without the coating. Thus, the tribological characteristics of the Ni–C–Ag–N coating formed by the complex method exceed by several times those of the copper as well as the characteristics of the Ni–Ag–N coating produced earlier.

The hardness of the coating was measured on a transverse metallographic section along three tracks running perpendicular to coating surface, which allowed the average hardness of coating to be determined and the dependence of hardness of material under study on distance from coating's surface to be detected. The results presented in Fig. 10 show that the hardness of the coating reaches its maximum value of 1780 MPa that is 1.4 times higher than that of a substrate in the adjacent layer. The hardness of the substrate layer adjacent to the coating increases that of a substrate bulk by 15% which is indicative of a substrate strengthening as a result of thermomechanical effect. Attention is paid to a low value of hardness







Fig. 16 – A structure of a copper transverse metallographic section with the Ni–C–Ag–N coating. b – energy spectra obtained from a portion of the coating designated on (a) with '+'sign. A table lists the results of quantitative analysis of the given coating portion.

of 'coating/substrate' transition layer which may be caused by the presence of micropores and tensile stresses in the layer. The substrate hardness amounts to 1300 MPa. If we recalculate the values to units of the hardness number HV then the hardness value of coating is 182 and that of the substrate (copper M00 grade) is 133. Form the literature it is known that the hardness of the Ag–Cu phase is not very high and is usually ≈ 100 HV [35]. The increased hardness value of the Ni–C–Ag–N system coating against the hardness of the Ag–Cu phase and the copper substrate is due to a composite structure of the coating formed by a copper matrix strengthened with high-strength high-module particles of carbonitrides, carbides and nitrides of silver and nickel. The thickness of the coating reaches 90 μ m (Fig. 11, *a*) as it was determined as a result of transverse metallographic sections' examination by the methods of scanning electron microscopy. It is clearly seen that the coating is structural and, in all likelihood, is nonuniform in phases. The coating contains inclusions of different shapes and sizes (Fig. 11, b). The sizes of inclusions of etched metallographic section revealed by the methods of scanning electron microscopy vary in the limits of $3 \mu m$.

The elemental composition of the coating was studied by the method of micro-X-ray-spectral analysis. It is established by the method of mapping of metallographic section that the elements being present in the coating are distributed quasinoniformly (Fig. 12).

A substantially nonuniform distribution of chemical elements in the coating (Fig. 13) is detected by the mapping method of transverse foil (STEM analysis). Namely, in a silverbase matrix the islands of copper and nickel are revealed. The



Fig. 17 – The Ni–C–Ag–N coating formed on copper, a – a bright field image, b – a microelectron diffraction pattern; c, d – dark field images obtained in reflections [222]Ag (c) μ [200]Cu + [310]AgN₃. (b) the arrows indicate the reflections in which dark field images are obtained: 1 – a dark field image (c), 2 – a dark field image (d).

sizes of the islands vary in the limits of $0.1-0.4 \mu m$. A concentration of chemical elements in foil portions (Fig. 13) under study is presented in Table 3 and Table 4.

The results presented in Table 3 indicate that silver is the main chemical element of the coating, copper being present in smaller quantity and nickel concentration being extremely small.

A relative content of chemical elements and their distribution in the coating is shown in Fig. 14. Results of the analysis correlate with the results presented in Fig. 15 and indicate, firstly, predominant concentration of silver in the coating, and, secondly, the alloying of the coating with copper atoms whose concentration increases with a greater distance from the coating surface.

Analysis of energy spectra obtained from points located at different distance from the coating surface enabled the elemental composition of the coating to be quantitatively assessed. It is determined that the main element of the coating is silver, nitrogen being present in a somewhat smaller quantity. The remaining chemical elements are present in the coating in a substantially smaller quantity.

Example of investigation into the elemental composition of the coating 'by points' is given in Fig. 16.

A phase composition of the coating and morphology of phases forming it were studied by the methods of transmission electron diffraction microscopy. The results obtained by the method are shown in Fig. 17. The obtained results indicate that silver has a polycrystalline (granular) structure (Fig. 17, c). In bulk of grains a dislocation substructure in the form of chaotically distributed dislocations is present. Particles of the second phase (copper and silver nitride) locate as particles in bulk and along boundaries of silver grains (Fig. 17, d). The results of electron microscope microdiffraction analysis presented in Fig. 18 demonstrate the presence of silver carbonitride particles (Fig. 18, d). The particles have a rounded shape and locate both in the bulk of silver grains and at their boundaries. Dimensions of silver carbonitride particles located in the bulk of grains vary in the limits of 10-30 nm; those located along grain boundaries – from 20 to 40 nm.

4. Conclusions

By means of a complex method combining the electroexplosive spraying, irradiation by pulsed electron beam and subsequent nitriding in plasma of low pressure gas discharge the Ni–C–Ag–N coatings \approx 90 µm thick have been formed. The wear resistance of a copper sample with the deposited coating is \approx 1.6 times higher than that of copper without the coating. The friction coefficient of samples with the coating (µ = 0.50) is

 \approx 1.35 times smaller than that of copper without the coating ($\mu = 0.679$). It has been detected that the hardness of the coating increases as substrate is approached and reaches its maximum value of \approx 1780 MPa (hardness of a substrate is 1300 MPa). By the methods of micro-X-ray-spectral analysis it has been found that silver is the main chemical element of the coating, copper, nickel, carbon and nitrogen being present in a considerably smaller quantity. By the methods of diffraction electron microscopy it has been established that the main phases of the coating are the copper and silver base solid solutions. The inclusions of silver nitrides AgN3 and carbonitrides AgCN of a nanometer range have been discovered. Based on the obtained results, we can conclude, that the increase in strength and tribological characteristics of the coating relative to the copper substrate is caused by the formation of a sub-micro-nanocrystalline multiphase structure. The Ni-C-Ag-N coating



Fig. 18 – The Ni–C–Ag–N coating formed on copper, a – a bright field image, b – a microelectron diffraction pattern; c, d – dark field images obtained in reflections [220]Ag (c) and [111]Ag + [111]AgCN. b – arrows indicate the reflections in which dark field images are obtained: 1 – a dark field image (c), 2 – a dark field image (d).

withstands 7000 on/off cycles in testing of electromagnetic starters for the commutation wear resistance in mode AC-3. The resistance of paired electrical contact in all three phases L1–L3 is less than 15 μ Ohm in the process of testing's. The time necessary for contacts to be disconnected is 20–25 μ s. The electrical conductivity of the coatings formed amounts to 62.0 MS/m. The mode of electroexplosive spraying at power density of \approx 5.5 GW/m² being absorbed and with the use of the exploded conductor based on silver, nickel and carbon should be considered to be the optimum one for the formation of coatings. After which electron beam processing is carried out with the following parameters: 17 keV, 20J/cm², 200 μ m, 5 pulses, 0.3s⁻¹ and nitriding at 600 °C for 3 h.

CRediT authorship contribution statement

Conceptualization - Denis Romanov, Vasilii Pochetukha. Investigation - Denis Romanov, Vasilii Pochetukha, Kirill Sosnin, Stanislav Moskovskii, and Victor Gromov. Methodology - Vladimir Bataev, Yurii Ivanov, and Alexander Semin; Project administration - Denis Romanov; Supervision — Alexander Semin. Validation - Denis Romanov, Vasilii Pochetukha, and Kirill Sosnin; Resources - Denis Romanov; Writing - original draft, all authors. Review and editing - Denis Romanov, Vasilii Pochetukha, Kirill Sosnin, Stanislav Moskovskii, Victor Gromov, and Vladimir Bataev.

All authors have read and agreed to the published version of the manuscript.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Denis A. Romanov reports financial support was provided by Siberian State Industrial University

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