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СТРУКТУРА ЭЛЕКТРОЭРОЗИОННОСТОЙКОГО КОМПОЗИЦИОННОГО ПОКРЫТИЯ СИСТЕМЫ TiB_2–Ni–Ag, ПОЛУЧЕННОГО МЕТОДОМ ЭЛЕКТРОВЗРЫВНОГО НАПЫЛЕНИЯ

Василий Витальевич Почетуха[1], Денис Анатольевич Романов[1†],
Екатерина Степанова Ващук[1,2], Екатерина Андреевна Мартемьянова[1],
Виктор Евгеньевич Громов[1]

[1]Сибирский государственный индустриальный университет, ул. Кирова, 42, 654006, Новокузнецк, Россия
[2] Филиал Кузбасского государственного университета им. Т.Ф. Горбачева, ул. Ноградская, 19а, 653039, Прокопьевск, Россия
v.pochetuha@mail.ru, https://orcid.org/0000-0003-0492-6188
romanov_da@physics.sibsiu.ru, https://orcid.org/0000-0002-6880-2849
vaschuk@bk.ru, https://orcid.org/0000-0002-1345-7419
martemianova_ea@sibsiu.ru, https://orcid.org/0009-0001-1882-2753
gromov@physics.sibsiu.ru, https://orcid.org/0000-0002-5147-5343

Аннотация. В настоящей статье обсуждаются вопросы формирования композиционного покрытия на основе диборида титана, серебра и никеля, а также исследования структуры покрытия на микрокристаллическом и нанокристаллическом уровне. Исследуемое покрытие получено на медной подложке за счет одновременного электрического взрыва серебряной и никелевой фольги, а также расположенным на поверхности фольг порошком диборида титана. Исследования структуры праведны с использованием методов сканирующей и просвечивающей электронной микроскопии, также исследована переходная область между покрытием и медной подложкой. Покрытие имеет среднюю толщину 160 мкм и характеризуется структурой с включениями диборида титана микрокристаллического и нанокристаллического размера, расположенными в матрице на основе серебра и никеля. Нанокристаллическая структура покрытия представляет собой твердые растворы на основе меди, никеля и серебра в которых располагаются наноразмерные частицы диборида титана. Локально установлена начальная стадия распада твердых растворов при образовании каких-либо других фаз. В переходной области между покрытием и подложкой нанокристаллическая структура покрытия также представляет собой наноразмерные частицы, представленные твердыми растворами на основе меди, серебра и никеля. Также титан, который образовался после частичного распада фазы диборида титана при электровзрывном напылении, входит в твердый раствор фазы на основе никеля. Фаза на основе серебра заполняет промежутки между фазой на основе никеля и титана. В переходной области также выявлены крупные частицы, практически полностью состоящие из диборида титана.

Ключевые слова*:* композиционное покрытие, электровзрывное напыление, микроструктура, наноструктура, диборид титана, никель, серебро, медная подложка, фаза, сканирующая электронная микроскопия, просвечивающая электронная микроскопия.

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Original article

STRUCTURE OF ELECTRO-EROSSION-RESISTANT COMPOSITE COATING OF THE TiB_2–Ni–Ag SYSTEM OBTAINED BY ELECTRO-EXPLOSIVE SPRAYING

Vasily V. Pochetukha[1], Denis A. Romanov[1†], Ekaterina S. Vashchuk[1,2], Ekaterina A. Martemianova[1], Viktor E. Gromov[1]

[1] Siberian State Industrial University, Kirova Ave., 42, 654007, Novokuznetsk, Russia
[2] Department of Economics and Natural Sciences, T.F. Gorbachev Kuzbass State Technical University, Prokopyevsk branch, Nogradskaya st., 19a 653039, Prokopyevsk, Russia
v.pochetuha@mail.ru, https://orcid.org/0000-0003-0492-6188
romanov_da@physics.sibsiu.ru, https://orcid.org/0000-0002-6880-2849
vaschuk@bk.ru, https://orcid.org/0000-0002-1345-7419
martemianova_ea@sibsiu.ru, https://orcid.org/0009-0001-1882-2753
gromov@physics.sibsiu.ru, https://orcid.org/0000-0002-5147-5343

Abstract. This article discusses the formation of a composite coating based on titanium diboride, silver and nickel, as well as studies of the coating structure at the microcrystalline and nanocrystalline levels. The coating under study was obtained on a copper substrate by simultaneous electrical explosion of silver and nickel foil, as well as titanium diboride powder located on the surface of the foils. The structure was studied using scanning and transmission electron microscopy, and the transition region between the coating and the copper substrate was also studied. The coating has an average thickness of 160 μm and is characterized by a structure with microcrystalline and nanocrystalline titanium diboride inclusions located in a silver- and nickel-based matrix. The nanocrystalline structure of the coating is solid solutions based on copper, nickel and silver in which nanosized particles of titanium diboride are located. The initial stage of decomposition of solid solutions during the formation of any other phases is locally established. In the transition region between the coating and the substrate, the nanocrystalline structure of the coating also represents nanosized particles represented by solid solutions based on copper, silver and nickel. Also, titanium which was formed after partial disintegration of the titanium diboride phase during electro-explosive spraying is included in the solid solution of the nickel-based phase. The silver-based phase fills the gaps between the nickel-based and titanium-based phases. Large particles consisting almost entirely of titanium diboride were also detected in the transition region.

Keywords: composite coating, electro-explosive spraying, microstructure, nanostructure, titanium diboride, nickel, silver, copper substrate, phase, scanning electron microscopy, transmission electron microscopy.

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Introduction

Electrical contacts are important components of electric switches in electric circuits that allow current to pass through a contact between two conductors. Electrical contacts are in charge of connecting, conducting and interrupting normal and fault current. Their quality and lifetime are directly related to the characteristics of a particular switching device of a particular electric circuit. Electrical contacts are mainly used in relays, contactors, air circuit breakers, current-limiting switches, motor protection devices, micro switches, protective instruments, computer keyboards, portable machines, home appliances, automotive equipment (window lifters, rear-view mirrors, light switches, motor starters and other load interrupter switches), leakage protection switches [1], etc. There are many materials for making electrical contacts, the following being mainly used for arc-resistant electrical contacts: silver, silver-nickel [2], silver-copper oxide, silver-cadmium oxide, silver-tin oxide, silver-tin oxide and indium oxide, silver-zinc oxide [3], red copper, brass, phosphor copper, bronze, tin copper, beryllium copper [4], copper-nickel alloys, zinc-nickel copper, stainless steel [5] and so on. However, modern metal contacts have problems such as chemical corrosion resistance, poor arc ablation resistance and high manufacturing costs, which limits their application [6]. In the atmosphere, switching components often generate sparks or arcs when connecting or breaking a circuit. The occurrence of an electric arc during switching of electrical contacts inevitably leads to oxidation and ablation of the contacts, and can also lead to carbonization of organic substances in the air with the formation of carbon deposits which will gradually increase the contact resistance of the switch or can lead to an open circuit. The main contacts of low-voltage contactors are usually made of a silver-nickel alloy or an alloy based on silver and tin oxides, with the silver content being more than 90%, and the rest is nickel or tin oxide [7]. Silver has good electrical conductivity which ensures low contact resistance and prevents overheating of the contacts while nickel or tin oxides have good resistance to arc ablation which increases the lifetime of the main contacts of the contactor.

The above-described electrical contact materials are obtained by bulk production methods, such as:

1. Liquid-phase methods for producing metal-matrix composite materials:

– method of mechanical mixing of reinforcing particles into the melt [8];

– blotting method [9].

2. Solid-phase methods for producing metal-matrix composite materials [10]:

– mechanical processing of powders in high-energy ball mills [11];

– methods for sintering metal-matrix composite materials [12];

– methods of powder metallurgy [13];

Methods for producing composite arc-resistant coatings on contacts of switches of powerful electrical networks include:

– surfacing methods [14];

– spraying methods (gas flame [15], plasma [16], detonation [17], high-speed gas flame [18], cold gas-dynamic [19] and electric explosive [20]).

The above-mentioned literature outlines the existing scientific problem of increasing the lifetime of electrical contact materials, as well as their restoration by forming new structures and phase compositions of bulk electrical contact materials or by forming arc-resistant coatings on the surface of electrical contacts.

The purpose of this research paper was to study the structure of the electrical erosion-resistant composite coating of the TiB_2–Ni–Ag system obtained by electro-explosive spraying.

Material and research methods

The coatings were sprayed onto samples of electrical copper grade M00 (chemical composition, mass %: Cu 99.99; Fe 0.001; Ni 0.001; S 0.001; P 0.001; As 0.001; Pb 0.001; Zn 0.001; Ag 0.001; O 0.0005; Sb 0.0005; Bi 0.0005; Sn 0.0005), having the shape of a parallelepiped with dimensions of 25x25x5 mm. Electro-explosive spraying was carried out using silver foil (chemical composition, mass %: Ag 99.9; Pb 0.003; Fe 0.035; Sb 0.002; Bi 0.002; Cu 0.058) weighing 400 mg and nickel foil (chemical composition, mass %: Ni 99.99; Co 0.004; Al 0.0006; Bi 0.0001; Fe 0.002; Cd 0.0003; Si 0.001; Mg 0.001; Mn 0.001) weighing 20 mg. Titanium diboride powder TiB2 (particle dispersion 1–5 μm) weighing 200 mg was placed on the surface of the foils. The process was carried out with a plasma action time on the sample surface of ~ 100 μs and an absorbed power density at the jet axis of ~ 5.5 GW/m^2. Electro-explosive spraying of coatings was carried out on an EESM (Electrical explosive spraying machine) electric blasting unit at Siberian State Industrial University (Novokuznetsk, Russia).

Analysis and determination of the coating microstructure, distribution of elements were carried out using a KYKY-EM 6900 scanning electron microscope (made in China). The microstructure of the samples was also analyzed using a JEOL JEM-2100 universal thermal-field transmission electron microscope (made in Japan) with Oxford Instruments INCA Energy hardware for X-ray microanalysis. To conduct studies using the transmission electron microscopy method, samples were prepared in the form of thin “cross-section” foils. For this purpose, workpieces measuring 4×1×1 mm were cut out from the original samples. The cut workpieces were mechanically ground to 160 μm and then glued with epoxy resin to the treated surfaces. Then the blanks were placed in the Ion Slicer EM-09100 IS ion thinning system and prepared at an accelerating voltage of 7 kV until perforation occurred in the gluing area. Thin areas suitable for transmission electron microscopy (TEM) studies were located along the edges of the resulting perforation.

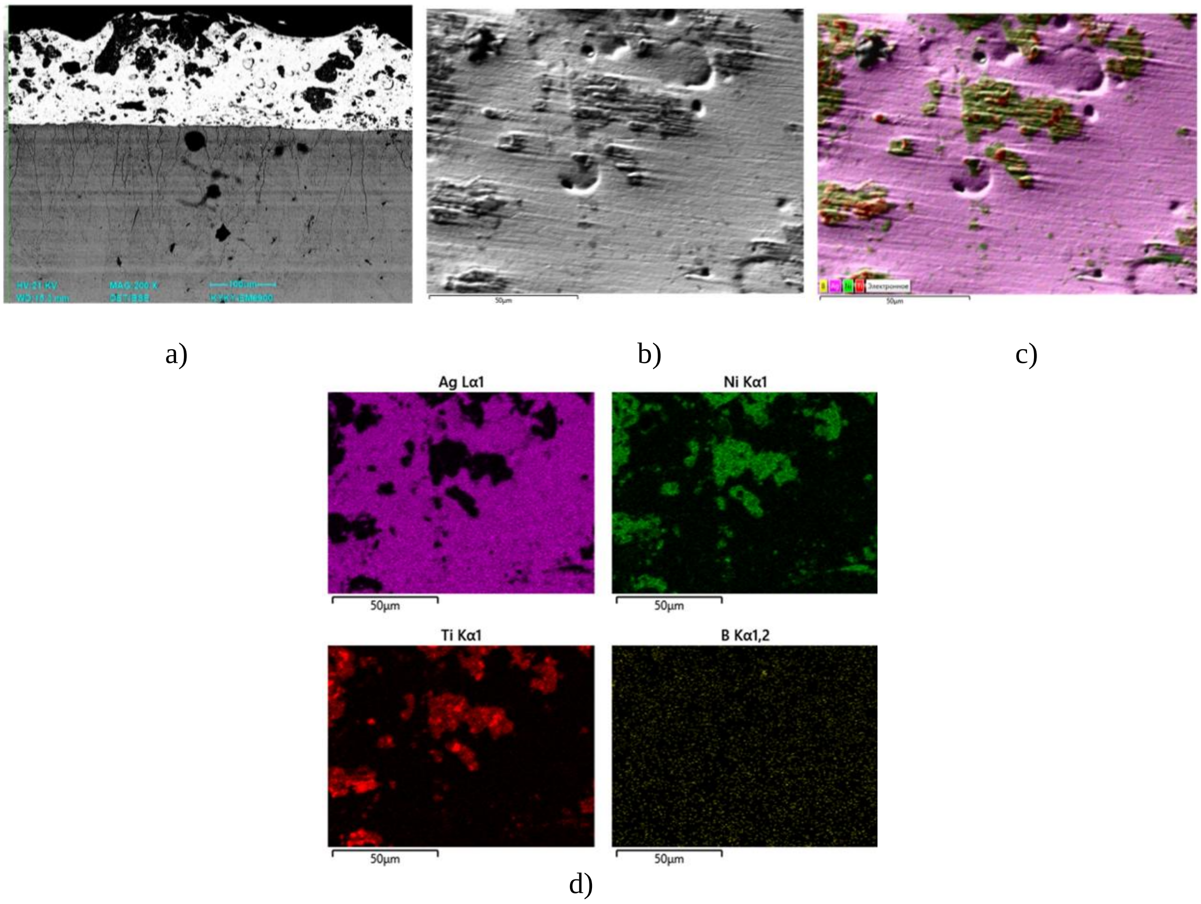
Results and discussion

The structure of the TiB_2-Ni-Ag coatings after electro-explosive deposition is represented by a silver matrix with inclusions of the strengthening phase consisting of TiB_2 and Ni (Fig. 1).

The average coating thickness is 160 μm. Their surface has a developed relief formed as a result of melt flows and hydrodynamic instabilities. At the boundary between the coating and the copper substrate there are periodic wave-like structures with micrometer-sized waves formed as a result of hydrodynamic instabilities during the coating application process.

The strengthening phase is represented by two types of inclusions. Large inclusions have an elongated shape. Their average length is 43 μm. It is worth noting that the largest elements can reach 100 μm in length. Their structure is represented by a nickel matrix with small inclusions of silver into which TiB_2 powder particles are fused. Large inclusions are located mainly in the upper and middle parts of the coating. Small inclusions with an average size of 8 μm are shifted closer to the boundary with the substrate. Elemental analysis showed that such inclusions consist of TiB_2 powder particles around which a thin nickel layer is formed.

In the copper substrate, cracks along the grain boundaries formed as a result of the impact of the shock-compressed layer extend from the interface with the coating.



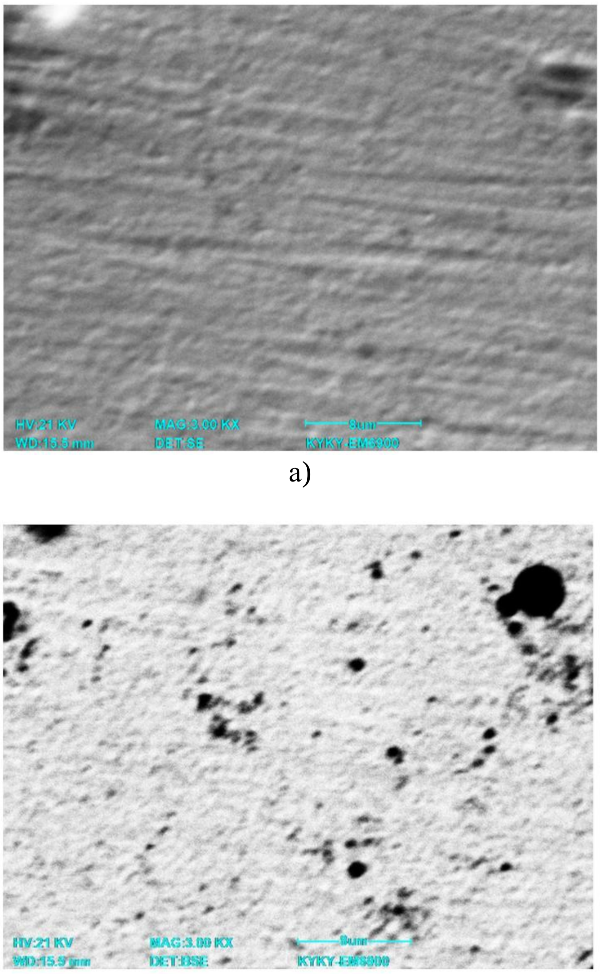
a) b) c)

d)

Рис. 1. СЭМ изображения структуры покрытия системы TiB_2–Ni–Ag, нанесенного электровзрывным методом, и медной подложки (а), серебряная матрица с включениями TiB_2–Ni (б) и карты распределения атомов Ag, Ti, B и Ni, полученные с использованием энергодисперсионной рентгеновской спектроскопии (в, г)

Fig. 1. SEM images of the structure of the TiB_2–Ni–Ag coating system deposited by the electro-explosive method and the copper substrate (a), the silver matrix with TiB_2–Ni inclusions (b) and the distribution maps of Ag, Ti, B and Ni atoms obtained using energy-dispersive X-ray spectroscopy (c, d)

The microstructure of the silver-nickel matrix is shown in Fig. 2. It contains many titanium diboride particles with an average size of 0.36 μm, formed as a result of nonequilibrium crystallization conditions. In areas with reduced gas solubility and cooling rate some particles consolidated into larger ones with a size of ~ 2 μm.



a)

b)

Рис. 2. СЭМ изображения микроструктуры матрицы системы TiB_2–Ni–Ag, полученного электровзрывным методом. (а) – SE режим сканирования; (б) – BSE режим сканирования

Fig. 2. SEM images of the microstructure of the matrix of the TiB_2–Ni–Ag system obtained by the electro-explosive method. (a) – SE scanning mode; (b) – BSE scanning mode

The diffraction pattern (Fig. 3) shows practically ring-shaped electron diffraction patterns represented by at least three fcc lattices. These reflections are probably produced by submicron particles being solid solutions based on Cu, Ni and Ag which is confirmed by the elemental analysis data. The bright-field (BF) and dark-field (DF) images show particles of various shapes (round, rectangular, irregular, in the form of shells) (Fig. 4).

When comparing the elemental analysis data (Fig. 5), the diffraction pattern (Fig. 4, d) and the TP images in reflections 9 (Fig. 4, b) and 11 (Fig. 4, c), the “gray” phase in the SP image (Fig. 4, a) including in the form of “shells” on rectangular particles belongs to the fcc solid solution based on Ag. While the “white” phase is the fcc solid solution based on Cu (the phases are indicated by the same colors as the element maps during mapping).

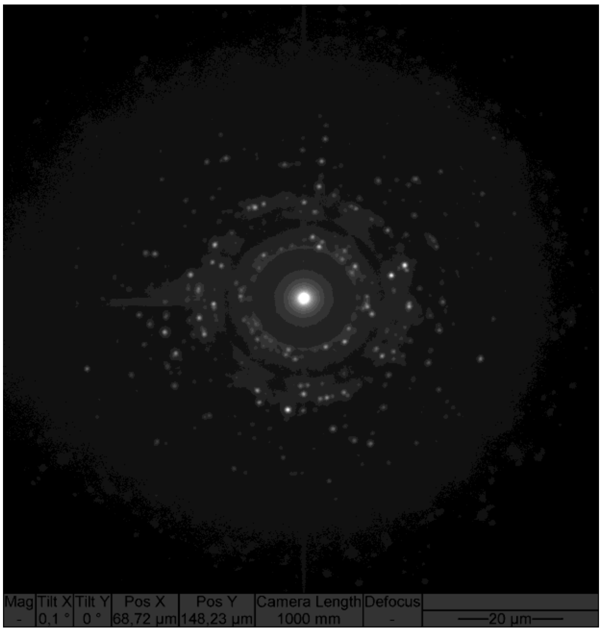
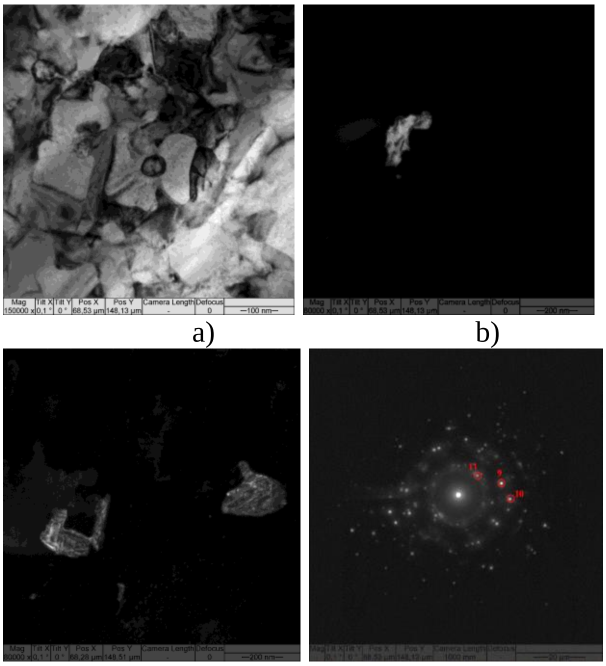


Рис. 3. Увеличенное изображение микродифракционной картины, полученной с участка покрытия системы TiB_2–Ni–Ag после электровзрывного напыления

Fig. 3. Enlarged image of the microdiffraction pattern obtained from a section of the TiB_2–Ni–Ag coating system after electro-explosive spraying

It should be borne in mind that the fcc solid solutions also contain Ni and Ti phases. Rounded nanosized particles are observed inside the rectangular particles, which are probably TiB_2 borides. The fact that the spectra from such particles in the elemental analysis differ slightly from the surrounding volume may be due to the fact that the final spectrum is provided by the volume located under the rounded particle. A detailed analysis of the diffraction pattern (Fig. 6) also reveals a Ni-based fcc phase (a Ni-based solid solution). The zone axis of this phase is close to [110]. In addition to this fcc phase, the microdiffraction pattern (MD) also contains reflections from another phase (probably fcc), which is not in a precise reflecting position.



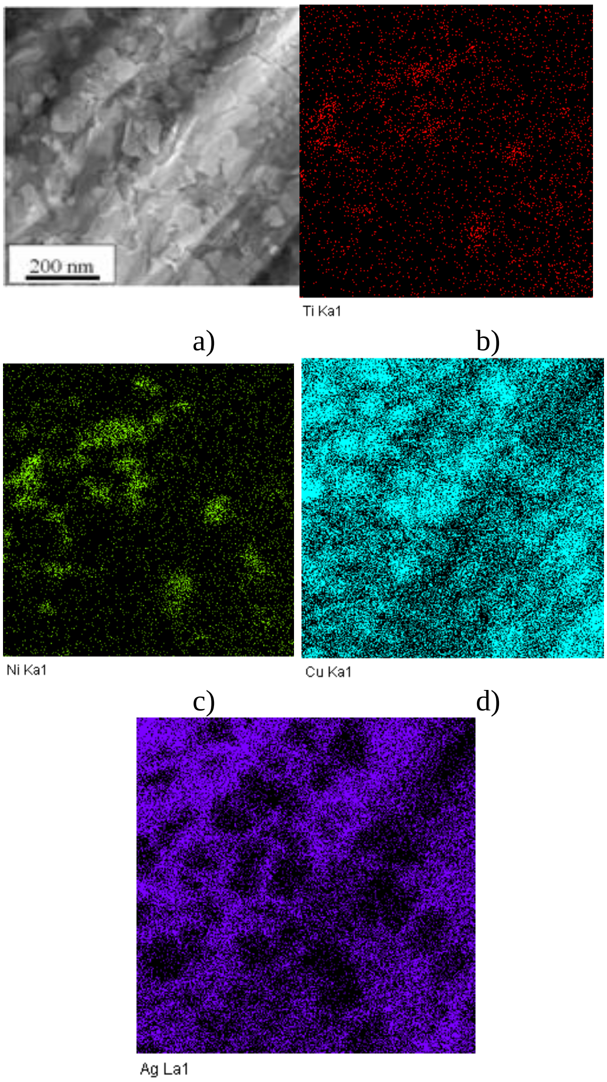
a) b)

c) d)

Рис. 4. Характерное светлопольное изображение (а) ПЭМ-структуры покрытия системы TiB_2–Ni–Ag после электровзрывного напыления и темнопольные изображения в рефлексах 9 (б) и 11 (в), а также микродифракционная картина (г)

Fig. 4. Typical bright-field image (a) of the TEM structure of the TiB_2–Ni–Ag coating system after electro-explosive spraying and dark-field images in reflections 9 (b) and 11 (c), as well as a microdiffraction pattern (d)

Figure 7 shows rounded nanosized particles embedded in the “matrix”. Dark-field analysis (Fig. 7) to the MD of Fig. 6 shows submicron particles of the “matrix” of the Ni-based fcc phase with a characteristic contrast within the particles. The contrast is associated with the initial stage of solid solution decomposition during the formation of some other phases. Judging from the results of elemental analysis (Fig. 8), large rounded particles are enriched in Ti and N, while other rounded particles are enriched in Cu. The areas containing the Ag-based solid solution generally do not coincide with the areas enriched in Ti, Ni and Cu.



a) b)

c) d)

e)

Рис. 5. Элементный анализ участка (а) к рис. 4, г в характеристических рентгеновских излучениях титана (б), никеля (в), меди (г) и серебра (д)

Fig. 5. Elemental analysis of section (a) in Fig. 4, g in characteristic X-rays of titanium (b), nickel (c), copper (d) and silver (d)

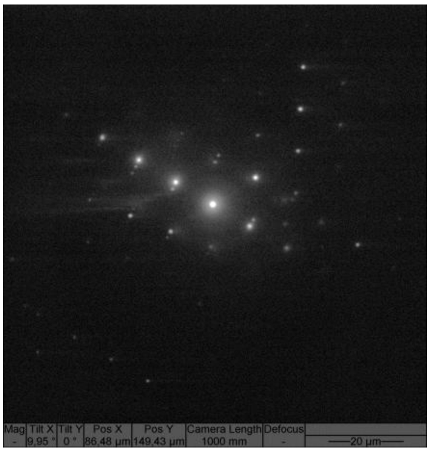
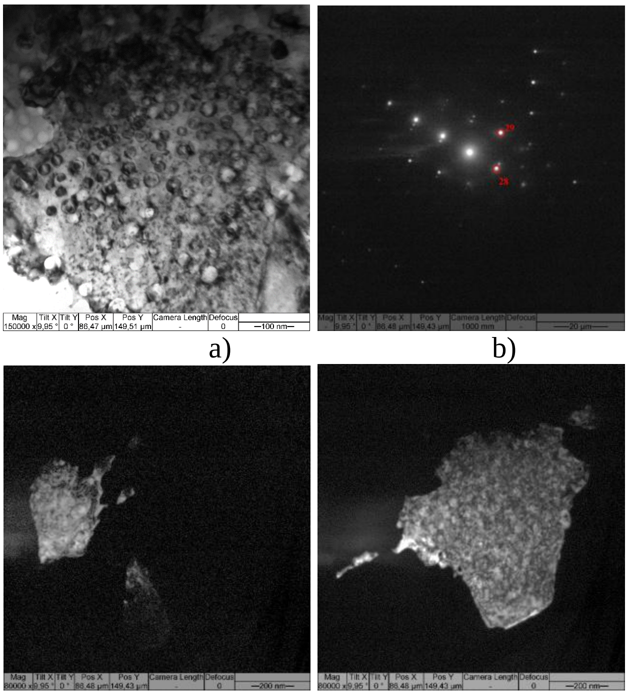


Рис. 6. Изображение микродифракционной картины, полученной с участка покрытия системы TiB_2–Ni–Ag и схема ее расшифровки

Fig. 6. Image of the microdiffraction pattern obtained from a section of the TiB_2–Ni–Ag system coating and a diagram of its interpretation

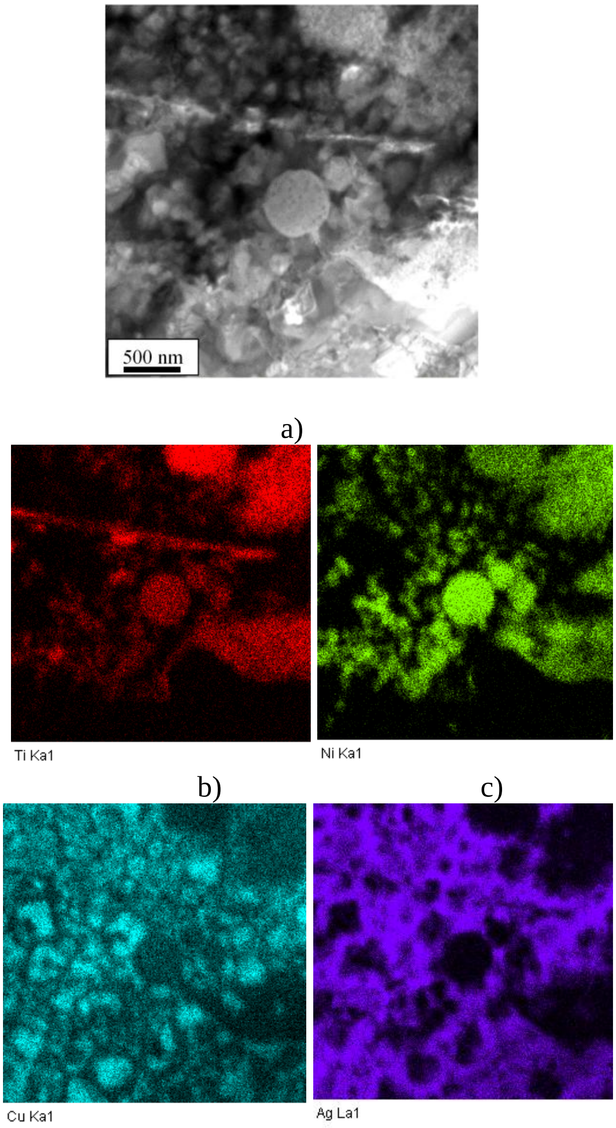


a) b)

c) d)

Рис. 7. Светлопольное изображение (а), микродифракционная картина (б) с отмеченными рефлексами и темнопольные изображения (с большим увеличением) в рефлексах (28 – в) и (29 – г), соответственно фазы на основе Ni

Fig. 7. Bright-field image (a), microdiffraction pattern (b) with marked reflections and dark-field images (with high magnification) in reflections (28 – c) and (29 – d), respectively, of the Ni-based phase



a)

b) c)

d) e)

Рис. 8. Элементный анализ увеличенного участка (а) к рис. 7. в характеристических рентгеновских излучениях титана (б), никеля (в), меди (г) и серебра (д)

Fig. 8. Elemental analysis of the enlarged section (a) of Fig. 7. in the characteristic X-ray radiation of titanium (b), nickel (c), copper (d) and silver (d)

Figure 9 shows a diffraction pattern with an analysis that allows the determination of fcc phases based on Cu, Ag and Ni.

The diffraction pattern was obtained from the transition region (Fig. 10) where the copper substrate is in the upper part of the frame, and nanosized particles represented by solid solutions based on Cu, Ag and Ni are in the lower part. Titanium is included in the solid solution of the nickel-based phase. The silver-based phase fills the gaps between the nickel-based and titanium-based phases.

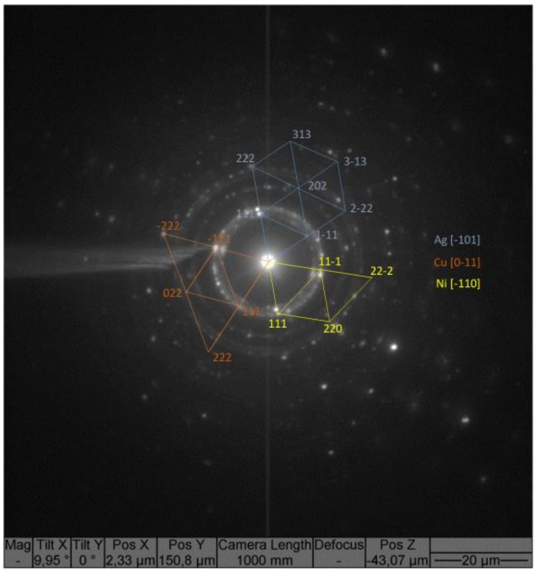


Рис. 9. Схема расшифровки микродифракционной картины от рис. 8

Fig. 9. Scheme for decoding the microdiffraction pattern from Fig. 8

In other areas of the foil, a structure is observed that differs in morphology from those presented above (Fig. 11, Table 1). On the right in this figure, there are large particles consisting almost entirely of titanium (boron is not shown due to a significant error in this element, spectra 4, 5, 8). The absence of extinction contours in these particles suggests that these particles are practically not deformed and are probably titanium-based borides (TiB_2). On the left, a structure similar to a "eutectic" is observed containing "dark" and "light" phases. The "dark" phase can be either separate relatively large particles or relatively thin plates in the "light" phase (pearlite-like structure). The "dark" phase is a solid solution of Ti-Ni-Cu. The "light" phase apparently has a different content of the main elements than the "dark" phase. Presumably, the left part of the image shows the decomposition of the solid solution with the formation of new phases. In addition, the diffraction patterns contain many reflections that may belong, among other things, to an intermetallic compound based on Ti-Ni-Cu.

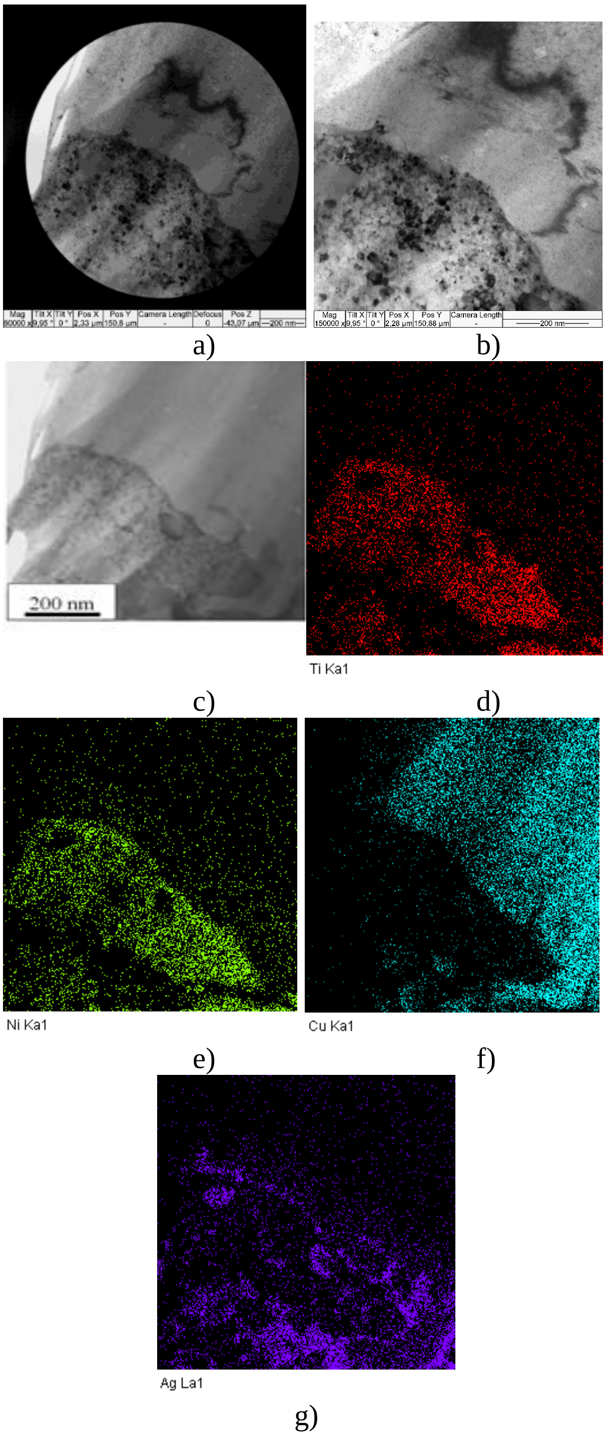


Рис. 10. Светлопольные изображения переходной зоны участка покрытия системы TiB_2–Ni–Ag после электровзрывного напыления (а–в) и элементный анализ (г–ж)

Fig. 10. Bright-field images of the transition zone of the TiB_2–Ni–Ag coating system after electro-explosive spraying (a–c) and elemental analysis (d–g)

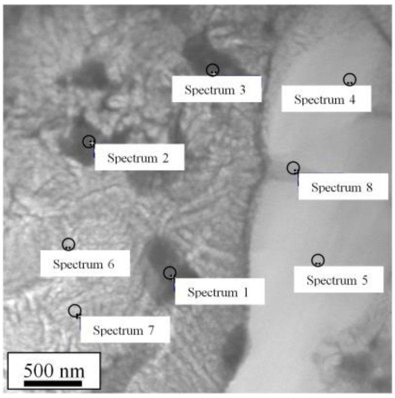


Рис. 11. STEM изображение с отмеченными точками элементного анализа

Fig. 11. STEM image with marked elemental analysis points

Таблица 1. Элементный анализ по точкам к рис. 11.

Table 1. Elemental analysis by points to Fig. 11.

Spectrum	B statistics.	Ti	Ni	Cu	Ag	Total
Spectrum 1	Yes	12.67	18.80	17.25	51.28	100
Spectrum 2	Yes	25.06	8.59	20.75	45.60	100
Spectrum 3	Yes	14.74	6.74	22.40	56.11	100
Spectrum 4	Yes	89.85	1.23	5.17	3.75	100
Spectrum 5	Yes	90.30	1.51	5.09	3.10	100
Spectrum 6	Yes	42.94	35.02	15.76	6.28	100
Spectrum 7	Yes	51.51	30.95	11.71	5.84	100
Spectrum 8	Yes	80.39	9.60	6.33	3.68	100

Conclusion

For the first time, a composite coating of the TiB2–Ni–Ag system was obtained by the electro-explosive spraying method and its structure was studied at the micro- and nanolevels. The coating has an average thickness of 160 μm and is characterized by a microstructure representing a silver matrix with inclusions of strengthening phases consisting of titanium diboride and nickel. The nanocrystalline structure of the coating represents solid solutions based on copper, nickel and silver, in which nanosized particles of titanium diboride are located. The initial stage of decomposition of solid solutions during the formation of any other phases was locally established. In the transition region between the coating and the substrate, the nanocrystalline structure of the coating also represents nanosized particles represented by solid solutions based on copper, silver and nickel. Also, titanium which was formed after partial decomposition of the titanium diboride phase during electro-explosive spraying is included in the solid solution of the nickel-based phase. The silver-based phase fills the gaps between the nickel- and titanium-based phases. Large particles consisting almost entirely of titanium were also detected in the transition region, and since boron was not determined due to a significant error in this element during TEM studies, as well as the absence of extinction contours in these particles, this proves that these particles are practically not deformed and are titanium diboride. In general, the formed structure with microcrystalline and nanocrystalline titanium diboride inclusions located in a silver- and nickel-based matrix suggests high mechanical properties and high electrical erosion resistance of the formed coatings.

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Информация об авторах

В.В. Почетуха – кандидат технических наук, старший научный сотрудник управления научных исследований Сибирского государственного индустриального университета.

Д.А. Романов – доктор технических наук, доцент, заведующий научной лабораторией электровзрывного напыления высоконадежных покрытий Сибирского государственного индустриального университета.

Е. С. Ващук – кандидант технических наук, старший научный сотрудник управления научных исследований Сибирского государственного индустриального университета, доцент кафедры естественнонаучных и экономических дисциплин Филиал Кузбасского государственного университета им. Т.Ф. Горбачева (г. Прокопьевск).

Е.А. Мартемьянова – кандидат филологических наук, доцент кафедры филологии Сибирского государственного индустриального университета.

В.Е. Громов – доктор физико-математических наук, профессор, заведующий кафедрой естественнонаучных дисциплин имени профессора В.М.Финкеля Сибирского государственного индустриального университета.

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Information about the authors

V.V. Pochetukha – Candidate of Technical Sciences, Senior Researcher of the Scientific Research Department of the Siberian State Industrial University, +7 (983) 216-86-88.

D.A. Romanov – Doctor of Technical Sciences, Associate Professor, Head of the Scientific Laboratory of Electro-explosive Spraying of Highly Reliable Coatings of the Siberian State Industrial University, +7 (952) 171-59-99.

E.S. Vashchuk – Candidate of Technical Sciences, Senior Researcher of the Scientific Research Department of the Siberian State Industrial University, Associate Professor of the Department of Natural Sciences and Economics of the Branch of the Kuzbass State University named after T.F. Gorbachev (Prokopyevsk, Russia), +7 (904) 378-01-14.

E.A. Martemyanova – Candidate of Philological Sciences, Associate Professor of the Department of Philology at the Siberian State Industrial University, +7 (951) 587-86-25.

V.E. Gromov – Doctor of Physical and Mathematical Sciences, Professor, Head of the Department of Natural Sciences named after Professor V.M. Finkel of the Siberian State Industrial University, , +7 (923) 636-19-82.

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