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THE USE OF POWDER MATERIALS OF HIGHLY HARD COMPOUNDS FOR THE FORMATION OF ELECTRIC SPARK COATINGS FOR VARIOUS FUNCTIONAL PURPOSES

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Abstract

Electric spark alloying of solid surfaces is a promising direction for increasing the performance properties of materials. Formation of superhard material coatings on a substrate allows to significantly increase hardness, wear resistance, resistance to high temperatures and pressure, and improve the performance characteristics of products by 1.5–5 times. This is due to the formation of various multicomponent structures in electric spark coatings that have increased strength and tribotechnical characteristics. The aim of the work was to study the structure and physical and mechanical properties of electric spark coatings obtained from powder materials. Various powder charge compositions and electric spark discharge parameters were used to form the coatings. The coatings were formed under standard climatic conditions by combining powder materials based on titanium carbide (TiC), aluminum (Al), carbon (technical graphite), titanium nitride (TiN), aluminum nitride (AlN) using the developed technology. The strength and adhesion properties of coatings obtained by the electric spark alloying method were studied. The studies to determine the adhesion characteristics using scratch analysis and Rockwell methods showed that coatings based on TiN+Al compounds have high adhesion strength. It was found that in TiN+Al coatings, electric spark alloying can lead to the formation of MAX phases and high-entropy compounds, which has a positive effect on the physical and mechanical properties of the formed coatings. The microhardness of the studied coatings is increased by 2–4 times compared to the original titanium substrates. The dependence of the coating microhardness on the indenter penetration depth was studied. The dependence of the strength characteristics on the indenter penetration depth of the TiC+Al electric spark coating (0.9 J) formed on the VT1 titanium alloy is nonlinear with an extremum in the region of a coating thickness of 9–10 μm. The strength characteristics of electrospark coatings formed by a contactless method from refractory metals were investigated. The possibility of forming coatings from silicate ceramics with increased values of microhardness and adhesive strength was established.

Keywords: electric spark alloying, hardness, nanocomposites, titanium carbides and nitrides.

ИСПОЛЬЗОВАНИЕ ПОРОШКОВЫХ МАТЕРИАЛОВ ВЫСОКОТВЕРДЫХ СОЕДИНЕНИЙ ДЛЯ ФОРМИРОВАНИЯ ЭЛЕКТРОИСКРОВЫХ ПОКРЫТИЙ РАЗЛИЧНОГО ФУНКЦИОНАЛЬНОГО НАЗНАЧЕНИЯ

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Реферат

Электроискровое легирование поверхностей твердых тел является перспективным направлением увеличения эксплуатационных свойств материалов. Формирование покрытий сверхтвердых материалов на субстрате позволяет существенно увеличить твердость, износостойкость, стойкость к воздействию высоких температур и давления, повысить эксплуатационные характеристики изделий в 1,5–5 раз. Это обусловлено образованием в электроискровых покрытиях различных многокомпонентных структур, обладающих повышенными прочностными и триботехническими характеристиками. Целью работы являлось изучение структуры и физико-механических свойств электроискровых покрытий, получаемых из порошковых материалов. Для формирования покрытий использовались различные составы порошковой шихты и параметры электроискрового разряда. Покрытия формировались в стандартных климатических условиях путем совмещения порошковых материалов на основе карбида титана (TiC), алюминия (Al), углерода (технического графита), нитрида титана (TiN), нитрида алюминия (AlN) по разработанной технологии. Проведены исследования прочностных и адгезионных свойств покрытий, полученных методом электроискрового легирования. Проведенные исследования по определению адгезионных характеристик методами скретч-анализа и

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Роквелла показали, что покрытия на основе соединений TiN+Al обладают высокой адгезионной прочностью. Установлено, что в покрытиях TiN+Al электроискровое легирование может приводить к образованию MAX-фаз и высокоэнтропийных соединений, что положительно сказывается на физико-механических свойствах формируемых покрытий. Микротвердость исследуемых покрытий повышена в 2–4 раза по сравнению с исходными титановыми подложками. Исследована зависимость микротвердости покрытия от глубины внедрения индентора. Зависимость прочностных характеристик от глубины внедрения индентора электроискрового покрытия TiC+Al (0,9 Дж), сформированного на титановом сплаве ВТ1, носит нелинейный характер с экстремумом в области толщины покрытия 9–10 мкм. Исследованы прочностные характеристики электроискровых покрытий, сформированных бесконтактным способом из тугоплавких металлов. Установлена возможность формирования покрытий из силикатной керамики, обладающих повышенными значениями микротвердости и адгезионной прочности.

Ключевые слова: электроискровое легирование, твердость, нанокомпозиты, карбиды и нитриды титана.

Introduction

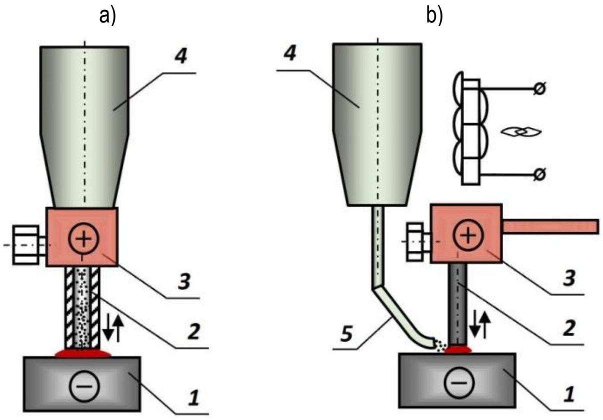
The electric spark alloying (ESA) method using powder materials is a promising technology for creating new-generation metallic materials and coatings with improved physical and mechanical properties. ESA of solid conductive surfaces involves the passage of electrode material between the electrodes, resulting in a directed ejection of the electrode material. The anode is predominantly destroyed during the electric spark discharge. Since ESA is carried out in a gaseous environment, this leads to the fact that under specified conditions, the anode material, which is mainly in the gas or liquid phase, is applied to the cathode. As a result of the interaction of the applied material with the cathode material and the environment, a layer with certain physical and mechanical properties is formed on the cathode. This layer has a complex chemical composition and structure and usually contains not only the anode material, but also solid solutions, chemical compounds, various alloys and pseudo-alloys. The formation of electric spark coatings leads to a significant change in the mechanical, electrical, thermal, magnetic, and thermionic properties of the modified surface layers of solids. The advantages of electric spark alloying are: high adhesive strength of the coating to the substrate; the possibility of obtaining coatings from refractory materials without heating the base material; the surfaces on which ESA coatings are formed do not require any preliminary preparation; simplicity, reliability and transportability of the process equipment [1–23].

Currently, there is insufficient information and knowledge about the main reasons for the limited life of tools and parts, factors that contribute to increased wear resistance, and a lack of understanding of the features of the electric spark process. In addition, there is insufficient practical experience in working with ESA installations using powder materials to form coatings. Also, for the successful use of this technological method of electric spark hardening, there is currently no necessary technological support for its application. With the high versatility of the electric spark method of applying metal nanocomposite coatings from powder materials, a system is required to simplify the methodology of their development to create effective hardening technologies. In this regard, the subject matter of the presented project is relevant. The main scientific idea is the use of powder materials of refractory metals and graphite with a certain percentage of ligands, which allow obtaining nanocomposite coatings of the appropriate composition and physical and mechanical properties during the process of electric spark alloying [6–9].

The aim of this work is to study the structure and physical and mechanical properties of electric spark coatings formed from powder materials of high-hardness compounds.

Experimental technique

Composite electric spark coatings based on nitrides, carbides, silicides of titanium and aluminum were applied by the method of electric spark alloying on a specialized installation (Figure 1), allowing the powder materials to enter the zone of electric spark discharge. Alloy VT1-0 was used as a substrate for the formation of coatings. The coatings were applied both to the metal in the delivery condition, which was ground to 9–10 purity class. Figure 1 shows the process flow chart for the formation of ESA coatings using this technology. To determine the optimal mode, in which the maximum amount of powder could get into the discharge zone, the vibration frequency of the processing electrode was slowly varied from 100 to 30 Hz. The ESA process was carried out in the range of discharge energy valuesfrom 0.3 to 10.0 J. Various powder charge compositions and electric spark discharge parameters were used to form the coatings (Table 1).



1 – workpiece (cathode); 2 – electrode (anode); 3 – applicator; 4 – hopper; 5 – powder feed tube into the working area; a – powder is introduced into the gap through a tubular electrode; b – powder is introduced from the side of the processing electrode

Figure 1 – Powder alloying methods

Table 1 – Coating compositions and modes of formation of electrospark coatings

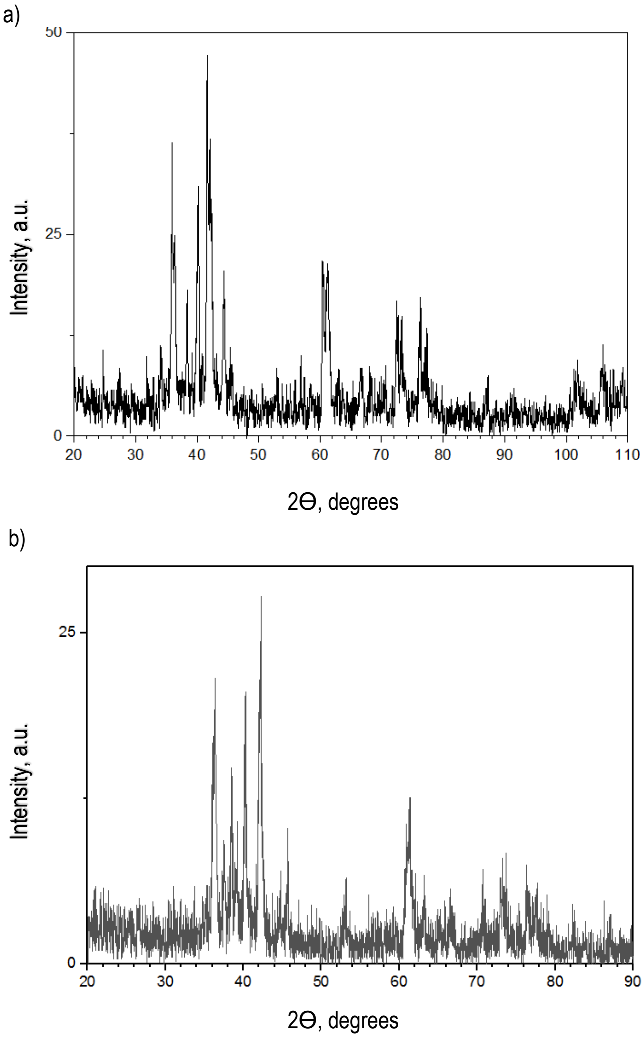
№	Composition of the powder mixture	Coating formation mode, J
1	–	–
2	TiC+Al	0.3
3	SiO_2	0.3
4	TiN	0.3
5	TiC+Al	0.9
6	Al+C	0.3
7	TiN+Al	0.3
8	Al+C	0.9
9	Ti+Al (heat treatment)	0.3
10	Ti (heat treatment)	0.3

The analysis of the structural features of metal coatings and their subjected to various types of processing was carried out on a universal metallographic complex manufactured by ZAO Spectroscopic Systems.

The features of the boundary layer structure in functional composite materials were studied using modern methods: scanning electron microscopy, atomic force microscopy, X-ray diffraction analysis (DRON-3.0) using standard techniques. X-ray diffraction analysis was used to determine the structure of thin-layer vacuum coatings. X-ray patterns were obtained on a general-purpose X-ray diffractometer DRON-3.0 using a standard technique, using the radiation of the Kα line from a tube with a copper anticathode, filtered at a wavelength of λ = 1.54051 Å. To measure the microhardness of coatings formed on metals, a hardness tester "Mikrosize uvicky" was used. The operating principle of the device is based on changing the linear value of the diagonal of the imprint c, obtained by pressing a diamond pyramid into the material under study under a certain load. A tetrahedral diamond pyramid with an angle of 136° between opposite faces was used for the studies, the load on the pyramid was 0.5 N. The thickness of the formed coatings was within ~ 60 μm.

Research results

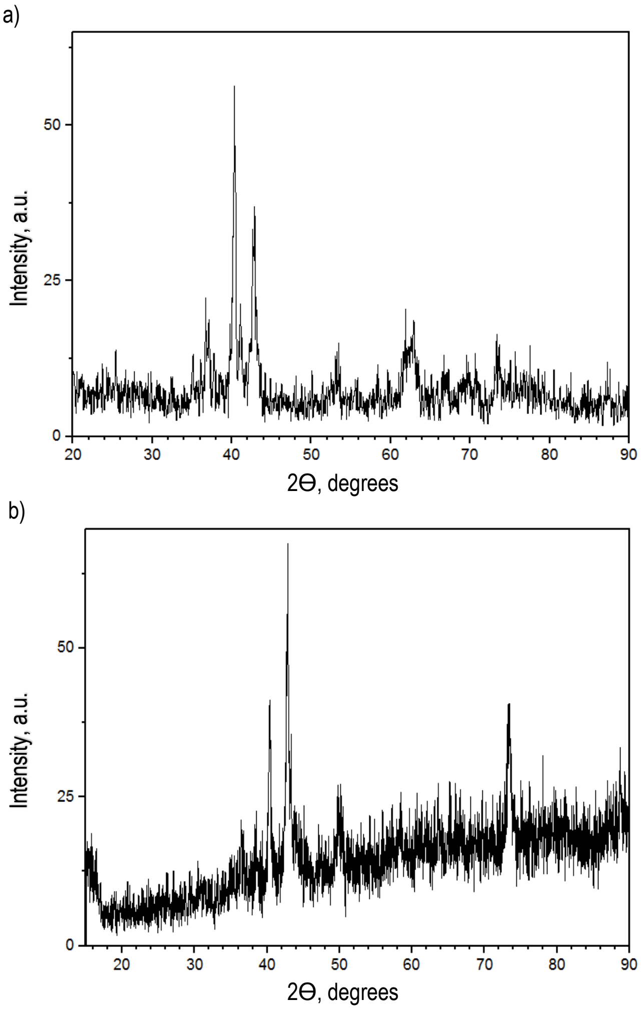
According to the X-ray phase analysis data, low-dimensional objects and MAX phases (Figures 2, 3) are possible in the electric spark coatings obtained by the contactless formation technology, which is confirmed by the presence of diffraction maxima in the areas of 2ϴ~38°, 53°, 61° (Figure 2a) and 2ϴ~43°, 73° (Figure 3b). The coatings were formed under standard environmental conditions by combining powder materials based on titanium carbide (TiC), aluminum (Al), carbon (technical graphite), titanium nitride (TiN), aluminum nitride (AlN) according to the technology shown in Figure 1.



a – TiC+Al coating, b – Al+C

Figure 2 – X-ray diffraction patterns of ESA coatings formed using a contactless method

In works [9–11] a qualitative method of determination of adhesive interaction between a solid substrate and a metal, ceramic coating obtained by various technological methods is proposed. The essence of the method lies in pressing an indenter in the form of a cone with an angle of 120° and a radius of curvature of the pin of 0.2 mm under a load of 150 kgf. Standard testing equipment is used for carrying out measurements, in particular, a Rockwell hardness tester. The holding time of the indenter in the coating under study is 6 s. After removing the load and extracting the indenter, the resulting imprint is studied using a metallographic optical microscope. The degree of adhesion of the coating is determined by studying the shape of the imprint. A variational series of images of the test results is constructed from good samples with a small number of cracks to samples in which complete peeling of the coating from the substrate is observed (Figure 4).



a – TiN coating, b – Ti+AlN

Figure 3 – X-ray diffraction patterns of ESA coatings formed using a contactless method

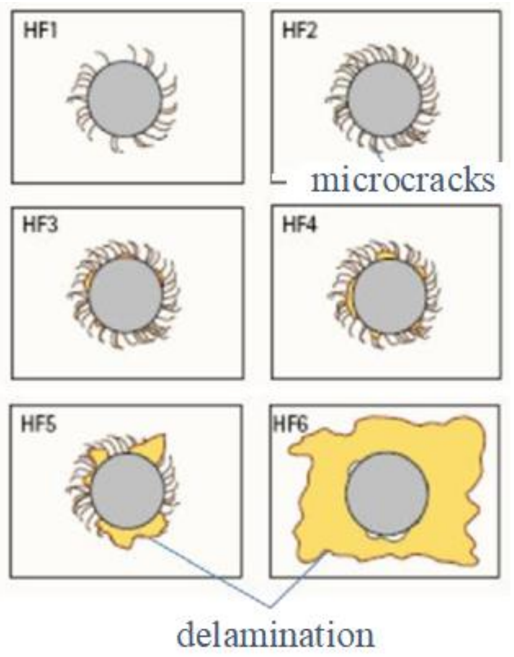
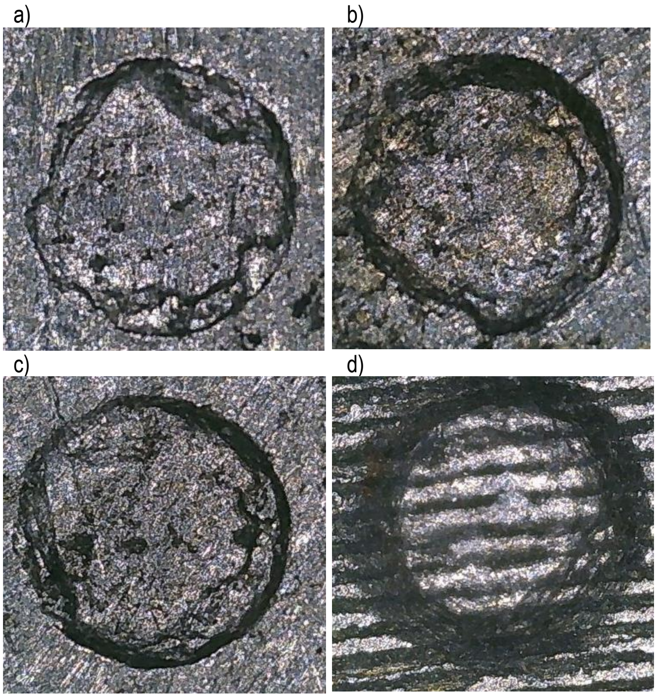


Figure 4 – Types of standardized coating failure prints according to DIN 4856:2018-02 and VDI 3198

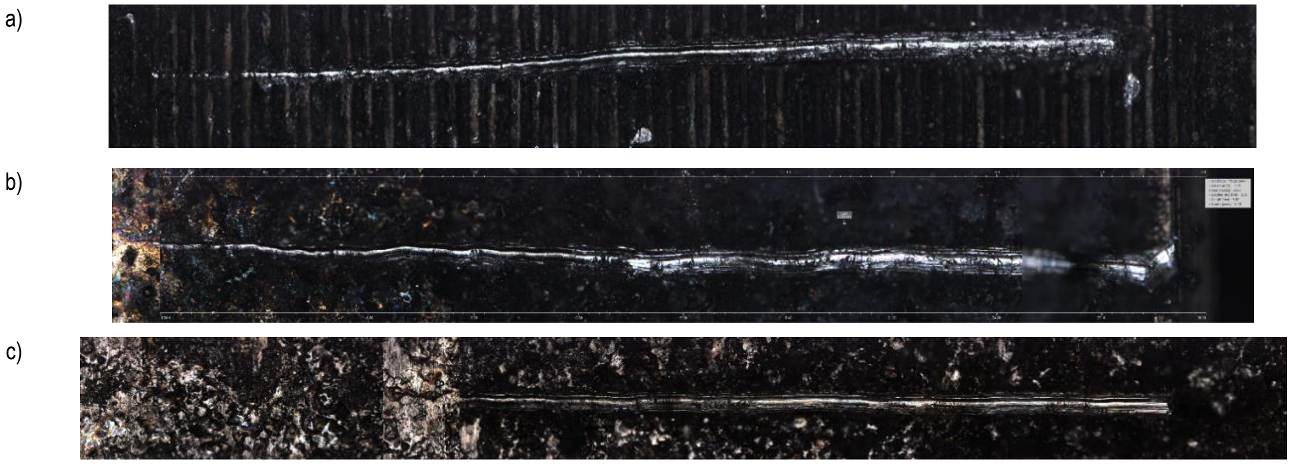
The results of the tests carried out according to DIN 4856:2018-02 and VDI 3198 are shown in Figure 5. According to the obtained data, the TiC+Al and SiO_2 electrospark coatings can be classified as HF5, the SiO_2 coating as HF4, and the TiN+Al coating as HF1 [12]. The high adhesive strength of coatings based on the TiN+Al system is most likely due to the fact that the use of aluminum allows increasing the plasticity of the coating while maintaining high strength characteristics. It is also possible to form compounds of the MAX phase type or high-entropy phases, since the substrate and coating contain the required amount of chemical elements to form these compounds, and the deposition process modes create the necessary physicochemical conditions for reactions that contribute to the formation of highly hard, plastic compounds. The scratch analysis method was used to determine the values of the adhesive interaction of the studied coatings sprayed by a contactless electrospark method onto metal substrates. The optimum mode, in which the highest values of adhesive interaction can be achieved, was determined with the following process parameters: the vibration frequency of the processing electrode was slowly varied from 100 to 30 Hz. Both industrial and experimental installations were used as sources of pulse discharges. The ESA process was carried out in the range of discharge energy values from 0.3 to 10.0 J. The results of the studies are presented in Figure 6.

The conducted studies to determine the microhardness values of electrospark coatings formed using contactless technology allowed us to establish an increase in the strength characteristics of modified titanium substrates by 1.3–5 times. The conducted studies to study the strength characteristics of electrospark coatings, in particular using the dynamic indentation method, showed an increase in the hardness values of titanium substrates after the formation of electrospark coatings obtained using contactless technology (Figure 8). To form the coatings, various powder charge compositions and electrospark discharge parameters were used (Table 1).



a – TiC+Al; b – SiO_2; c – TiN; d – TiN+Al

Figure 5 – Surface morphology of spark-ignition coatings after testing for adhesion strength according to DIN 4856:2018-02



a) Al+C; b)TiC+Al; c) Ti+AlN

Figure 6 – Morphology of the indentation surface during scratch analysis of the coating obtained by non-contact electric spark alloying

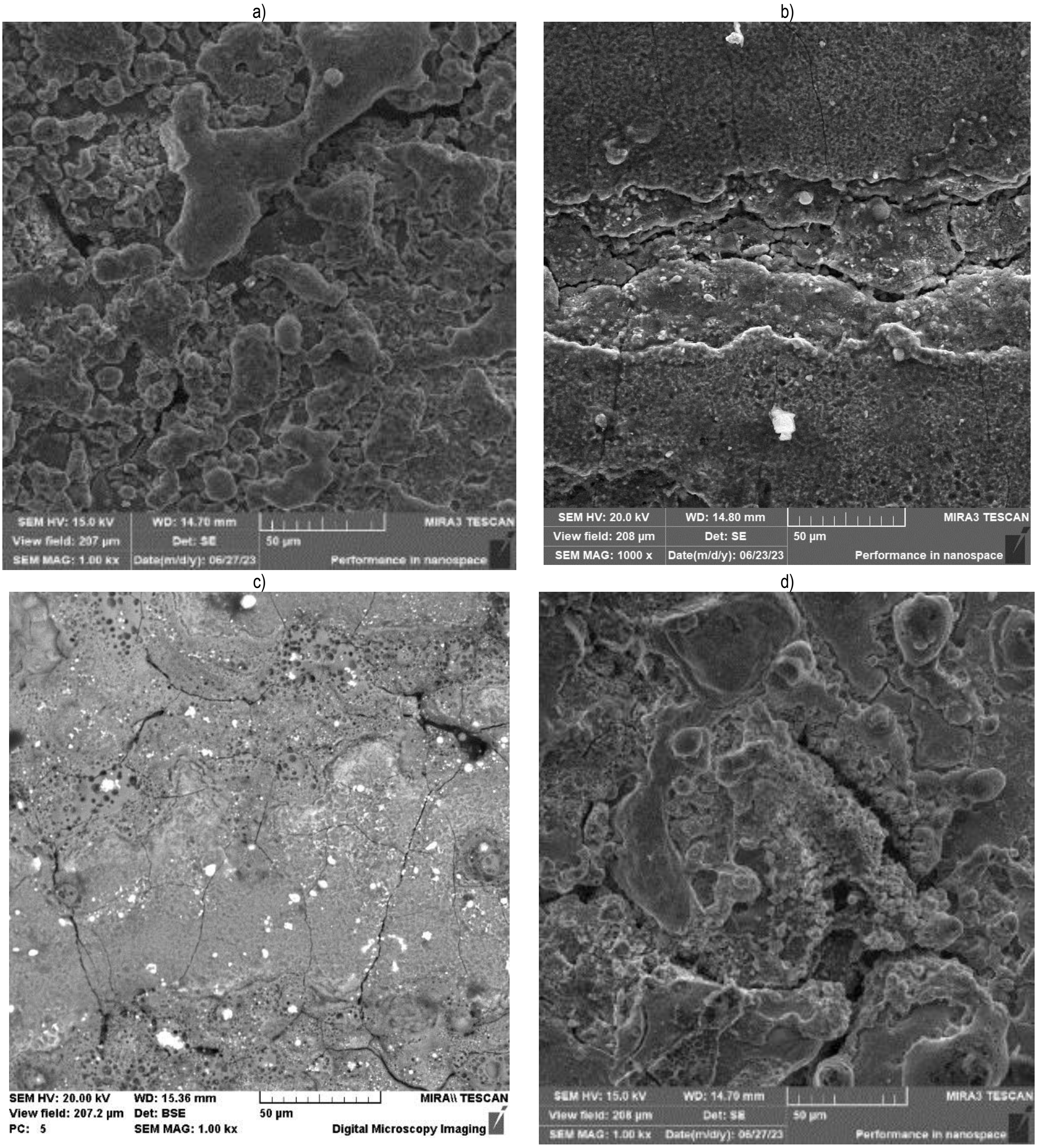
According to the data presented in Figures 4–6, it is evident that the highest adhesive strength is possessed by Ti+AlN coatings formed by the contactless method of electrospark alloying. Actual partial peeling of the coating is observed at normal load values in the region above 20 N. Whereas for Al+C, TiC+Al coatings, the onset of loss of adhesive strength begins at values of 10–15 N. Sufficiently high values of adhesive strength for Ti+AlN-based coatings are due to the application of a thermodynamically compatible titanium sublayer to the VT1 alloy by the electrospark method, which ensures good diffusion of the sublayer into the metal base.

Further deposition of the AlN-based coating, which is actually a ceramic and dielectric material, leads to the formation of surface layers with high physical and mechanical properties. High adhesive strength of the AlN layer to the metallic titanium sublayer is ensured by the fact that the entire process of forming this composite electrospark coating takes place in a single technological cycle. The results of determining the values of the adhesion work of the coatings to the titanium substrate correlate well with the scratch analysis data. The morphology of the electrospark coatings is quite developed. On the surface of the formed layers of refractory metals, a certain number of microroughnesses and voids are observed, which can be identified as closed pores and protrusions, as well as cracks (Figure 7).

The interest in the use of dynamic indentation to determine the hardness of materials is due to the fact that this approach allows us to determine the hardness under dynamic effects that constantly occur when using products in real operating conditions. While the methods of hardness testing (it is possible to consider as a special case of strength) according to Brinell, Rockwell, Vickers determine hardness statically, which in some cases does not provide complete information on the strength characteristics of materials used in structures that are operated under dynamic conditions. Determination of hardness values by the Leib method (dynamic indentation) is carried out according to the formula:

$$HL = 1000(v_b/v_a), \qquad (1)$$

where v_a is the speed of the spherical indenter falling before interacting with the surface of the measured material, v_b is the speed of the spherical indenter rebound after interacting with the surface of the studied material.

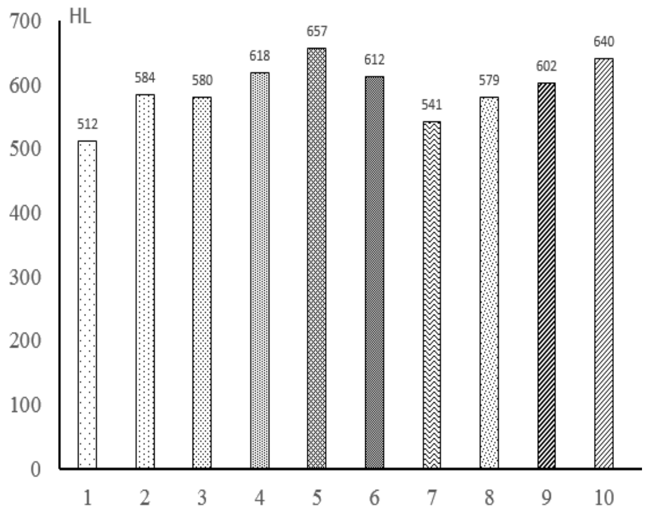


a – TiC+Al, b – Al+C, c – TiN, d – TiN+Al

Figure 7 – Morphology of ESA coatings

Based on the conducted studies, it is evident that the Leyd hardness values increased from 6 to 22 % compared to the original titanium material. Taking into account that the method of electric spark alloying is a surface hardening technology with a hardening zone thickness of approximately 40–60 µm, and the method of dynamic indentation leaves an imprint from the indenter with a depth of about 100 µm or more, the obtained hardness values indicate a significant modification of the surface layers of metal substrates when applying superhard coatings by the method of contactless electric spark alloying. The conducted studies of hardness by the Vickers microindentation method confirm the results obtained by the dynamic indentation method. Studies were conducted to study the microhardness of the titanium substrate depending on the depth of indenter (Figure 9). According to the obtained results, the microhardness of the surface layers of the original titanium (VT1 alloy) is not a constant value and changes depending on the depth of indenter penetration into the material under study. This dependence of the strength characteristics on the thickness of the surface layer coincides well with the theoretical and practical results associated with the structure of the surface layers of a solid.

The conducted studies on the microhardness of electric spark coatings formed on a titanium substrate made of VT1 alloy show an increase in the microhardness values for all types of formed ESA coatings (Figures 10, 11).



1 – original substrate; 2 – coating No. 2; 3 – coating No. 3; 4 – coating No. 4; 5 – coating No. 5; 6 – coating No. 6; 7 – coating No. 7; 8 – coating No. 8; 9 – coating No. 9; 10 – coating No. 10 (Table 1)

Figure 8 – Hardness values of electrospark coatings formed using contactless technology on titanium substrates

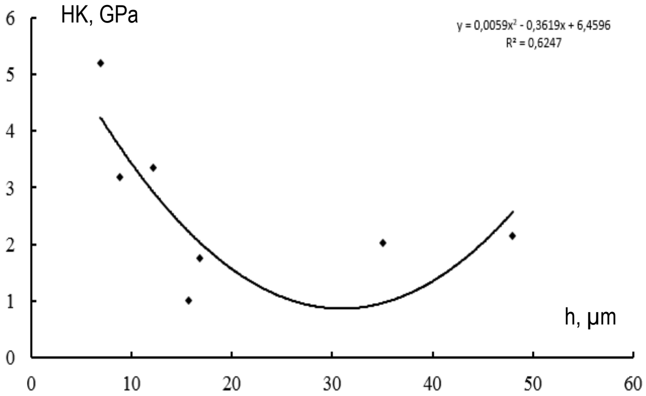
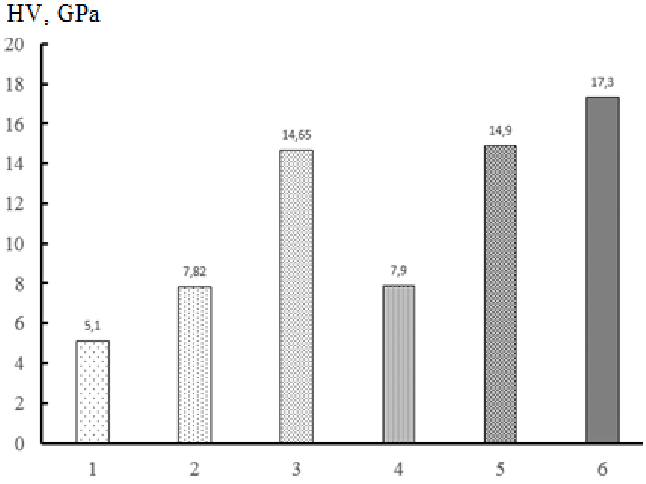


Figure 9 – Dependence of the microhardness of the titanium alloy VT1 on the depth of indenter penetration



1 – VT 1 (original sample); 2 – TiC+Al (0.3 J); 3 – SiO_2 (0.3 J); 4 – Al+C (0.3 J); 5 – Al+C (0.9 J); 6 – Al+C (1.2 J)

Figure 10 – Microhardness values of electrospark coatings formed by contactless technology on titanium substrates

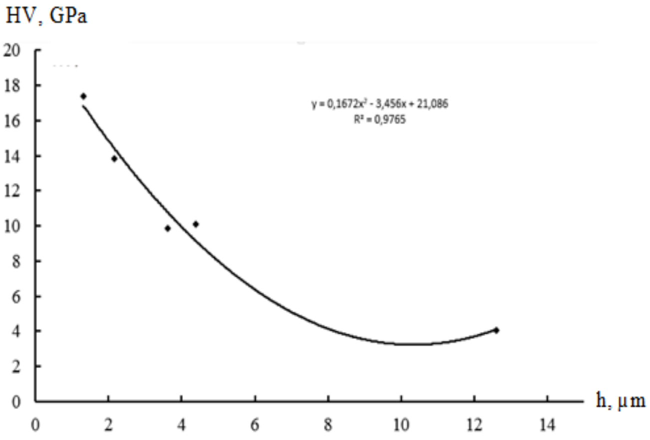


Figure 11 – Dependence of the microhardness of the TiC+Al (0.9 J) electric spark coating formed on the VT1 titanium alloy on the depth of indenter penetration

The dependence of the strength characteristics on the indenter penetration depth, as well as in the case of the control sample, is nonlinear. An extreme point is observed in the region of a coating thickness of 9–10 μm. The strength characteristics of electrospark coatings formed by a contactless method from refractory metals were studied.

Conclusion

The conducted studies have shown that the coatings formed by the ESA method have increased strength and adhesive properties. This effect is due to high values of the physical and mechanical properties of the deposited metals and alloys. The conducted studies to determine the adhesion characteristics using scratch analysis and Rockwell methods have shown that coatings based on TiN+Al compounds are characterized by high values of adhesive strength. This effect is explained by the thermodynamic compatibility of the applied spark coating and the titanium substrate, which ensures good diffusion of the sublayer into the metal base. In TiN+Al coatings, spark deposition can form MAX-phases and high-entropy compounds, which has a positive effect on the physical and mechanical properties of the formed coatings. It is shown that the microhardness values of the studied coatings exceed the values of the original titanium substrates by 2–4 times. The microhardness values depend on the composition of the applied powder materials, as well as the energy of the electric discharge in the area of obtaining the superhard coating.

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