ORIGINAL STUDY

THE EFFECT OF LAYERING ON THE PROPERTIES OF METAL-CERAMIC COATINGS OBTAINED BY DETONATION SPRAYING

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Abstract. *This work presents the results of a study of multilayer metal-ceramic coatings based on the NiCr/NiCr-Al_2O_3/ Al_2O_3 system obtained by detonation spraying. Coatings with different numbers of layers were obtained: three-layer (NiCr, 50% NiCr - 50% Al_2O_3, Al_2O_3), five-layer (NiCr, 75% NiCr - 25% Al_2O_3, 50% NiCr - 50% Al_2O_3, 25% NiCr -75% Al_2O_3, Al_2O_3) and six-layer (NiCr, Al_2O_3, NiCr, Al_2O_3, NiCr, Al_2O_3). The study aims to establish the influence of layering and gradient distribution of components on the microstructural, phase, mechanical, and tribological characteristics of coatings. The microstructural features of the coatings were studied using scanning electron microscopy in backscattered electron (BSE) mode and energy dispersive spectral analysis (EDS). The phase composition was analyzed by X-ray phase analysis. Microhardness and surface roughness measurements were performed, as well as tribological tests at a temperature of 700 °C. It was found that five-layer coatings with a gradient structure with a gradual transition from a metal layer to a ceramic layer demonstrate the best performance characteristics among all the samples studied. The five-layer coatings were characterized by high microhardness, uniform surface roughness, minimal counterbody penetration depth, and a stable friction coefficient in wear tests.*

Keywords: multilayer coating, metal ceramics, NiCr/NiCr- Al_2O_3/ Al_2O_3, detonation spraying, gradient structure, microstructure, X-ray phase analysis, tribological properties.

1. Introduction

The development of high-temperature anti-friction materials with low friction coefficients and high wear resistance is one of the key tasks for modern mechanical engineering, energy, and aerospace industries [1-3]. When operating under high mechanical and thermal loads, it is necessary to create coatings that can maintain stable performance characteristics over a long period of time, protecting parts from wear and thermomechanical damage. Among the various classes of materials, metal-ceramic coatings attract particular attention due to their combination of high mechanical properties, excellent wear resistance, and thermal and chemical stability when operating at high temperatures. These properties make metal-ceramic coatings promising candidates for the creation of high-temperature anti-friction systems [4-6].

Nickel (Ni) and nickel-chromium (NiCr) alloys are widely used as underlay materials in multilayer coatings due to their high heat resistance, corrosion resistance, and good adhesion to metal substrates [7]. The use of NiCr as a transition layer helps to effectively compensate for thermal stresses between the metal base and ceramic coatings, increasing the durability of the systems. One effective way to increase the wear resistance of coatings is to introduce ceramic phases such as aluminum oxide (Al_2O_3) [8]. Due to its high hardness, chemical inertness, and thermal stability, aluminum oxide has proven itself to be a promising component for creating tribological coatings that operate at elevated temperatures [9-11]. Earlier, Feng Liu and co-authors [12] investigated the mechanical and tribological properties of NiCr- Al_2O_3 composites at elevated temperatures. Their results confirmed the effectiveness of such systems for use in high-temperature friction conditions. However, most studies focused mainly on homogeneous composite materials. Unlike

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previous works, this study focuses on obtaining a gradient multilayer structure based on NiCr/NiCr-Al_2O_3/Al_2O_3. Gradient coatings provide a gradual change in composition between layers, which effectively reduces interlayer stresses arising from differences in the thermal and mechanical properties of the metal and ceramic phases. This approach improves adhesion between layers, increases crack resistance, and extends the service life of coatings during high-temperature operation [13,14].

The detonation spraying method is an effective technology for producing multilayer coatings with high density, low porosity, and high adhesion between layers. Due to the high particle velocity and rapid cooling, coatings with a specified structure and phase composition can be obtained. Of particular relevance is the creation of gradient multilayer systems, in which a gradual change in composition between layers reduces interlayer stresses and improves performance characteristics [15-17].

In this regard, the aim of this work is to obtain and study multilayer metal-ceramic coatings based on NiCr/NiCr-Al_2O_3/Al_2O_3, obtained by detonation spraying, with different numbers of layers (three-layer, five-layer, and six-layer structures). Particular attention is paid to analyzing the influence of the coating structure on the microstructural, phase, mechanical, and tribological properties during high-temperature operation.

2. Materials and methods

To conduct experiments on obtaining metal-ceramic coatings by detonation spraying, six samples were prepared from 12Kh18N10T steel rod (analogous to AISI 321; chemical composition, wt.%: Fe - base, Ni - 10, Cr - 18, Ti - 0.5, C - 0.12, Mn <2). The blanks were discs with a diameter of 50 mm and a thickness of 3 mm. Before applying the coatings, the surface of the 12Kh18N10T substrates was mechanically polished to ensure an average roughness Ra of less than 0.7 μm.

NiCr powder (Amperit 250.001) containing 80 wt% Ni and 20 wt% Cr with a particle size of about 63 μm (supplier - VIRS LLC, Kiev, Ukraine) was used as the metal component. White electrocorundum Al_2O_3 (grade 25A M40 Super, 87 wt% Al_2O_3, 13 wt% TiO_2, fraction 34 ±6 μm) supplied by INOKS LLC (Novosibirsk, Russia) was used as the ceramic component. The powder mixture was activated in a PULVERISETTE 23 planetary ball mill (FRITSCH, Germany). The mechanical activation time was 2 hours at a frequency of 30 Hz.

Multilayer metal-ceramic coatings based on NiCr/NiCr-Al_2O_3/Al_2O_3 were obtained by detonation spraying using the CCDS2000 installation (LICh SB RAS, Novosibirsk, Russia) [18]. Acetylene and oxygen were used as the combustible mixture during the spraying process. The Al_2O_3-based ceramic layer was sprayed at a gas mixture ratio of $O_2/C_2H_2 = 1.856$, with the detonation apparatus barrel filled to 63% with the explosive gas mixture. To obtain a NiCr metal layer, a gas ratio of $O_2/C_2H_2 = 1.063$ was used with a barrel filling degree of 54%. During the experiment, coatings with different numbers of layers were obtained: three-layer, five-layer, and six-layer.

The three-layer structure was obtained as follows: a NiCr metal layer was applied as the first layer to a pre-prepared surface of a 12Kh18N10T steel substrate; the second layer was a composite layer obtained by spraying a mixture of NiCr and Al_2O_3 powders in a mass ratio of 50:50; the final (third) layer was a ceramic layer of Al_2O_3. The five-layer coatings had a gradient structure, including the sequential application of the following layers: a NiCr metal layer, followed by three transitional composite layers obtained from powder mixtures of NiCr and Al_2O_3 with varying component ratios (75:25, 50:50, and 25:75 wt%), and, as the outer (fifth) layer, a ceramic layer of Al_2O_3. This gradient transition from the metal to the ceramic phase made it possible to reduce interlayer stresses and increase the adhesion of the coating. Six-layer coatings were created by sequentially alternating metal (NiCr) and ceramic (Al_2O_3) layers. The parameters of the technological mode for applying multilayer metal-ceramic coatings of the NiCr/NiCr-Al_2O_3/Al_2O_3 system are presented in Table 1.

Table 1. Technological parameters for obtaining multilayer gradient coatings based on NiCr/NiCr- Al_2O_3/ Al_2O_3.

Coating	Sprayed layers	Barrel filling volume, %	Number of shots
Three-layer	Al_2O_3	63	20
	NiCr(50%)-Al_2O_3(50%)	54	20
	NiCr	63	20
	Substrate	-	-

Table 1. (continued)

Coating	Sprayed layers	Barrel filling volume, %	Number of shots
Five-layer	Al_2O_3	63	10
	NiCr(25)-Al_2O_3(75)	63	10
	NiCr(50)-Al_2O_3(50)	54	10
	NiCr(75)-Al_2O_3(25)	54	10
	NiCr	54	10
	Substrate	-	-
Six-layer	Al_2O_3	63	5
	NiCr	54	5
	Al_2O_3	63	5
	NiCr	54	5
	Al_2O_3	63	5
	NiCr	54	5
	Substrate	-	-

The phase composition of multilayer metal-ceramic coatings was studied by X-ray diffraction using an X'Pert Pro diffractometer (Philips Corporation, Amsterdam, The Netherlands) in 2θ angle scanning mode in the range from 20° to 90°.

A TESCAN MIRA3 LMH scanning electron microscope (TESCAN, Brno, Czech Republic) equipped with an INCA ENERGY energy dispersive analysis attachment (Oxford Instruments, UK) was used to study the microstructure of multilayer metal-ceramic coatings.

Surface roughness (Ra) was determined using a 130 profilometer in accordance with GOST 25142-82. When determining the roughness of coatings, a speed of 0.25 mm/s, a measuring scale of 500 µm, and a travel distance of 5 mm were selected.

The microhardness of the coatings was determined using the Vickers method in accordance with GOST 9450-76 (ASTM E384-11) using a Metolab 502 microhardness tester (Metolab, Russia), with an indenter load of 0.1 N and an exposure time of 10 s.

High-temperature testing of tribological properties was carried out using a THT800 Anton Paar tribometer at a temperature of 700°C. During the tests, a load of 8 N, a sapphire ball with a diameter of 6 mm as a counterbody, and a linear sliding speed of 10 cm/s were used.

3. Results and discussion

The microstructural features of multilayer metal-ceramic coatings obtained by detonation spraying were investigated using cross sections with scanning electron microscopy in backscattered electron (BSE) mode, as well as elemental mapping using energy dispersive spectral analysis (EDS). Fig. 1 shows the obtained microstructure images illustrating the structure of coatings with different layer thicknesses (three-layer, five-layer, and six-layer structures).

Fig. 1a shows a cross-section of a three-layer metal-ceramic coating obtained by detonation spraying. Analysis of the microstructure shows clearly defined boundaries between the layers: metal (NiCr), composite (50% NiCr - 50% Al_2O_3), and ceramic (Al_2O_3). The resulting structure is characterized by density and the absence of defects in the form of pores and delamination, which indicates the high efficiency of the detonation spraying process. Fig. 1b shows the microstructure of a cross-section of a five-layer metal-ceramic coating (NiCr, 75% NiCr - 25% Al_2O_3, 50% NiCr - 50% Al_2O_3, 25% NiCr - 75% Al_2O_3, Al_2O_3). The structure of the coating demonstrates a gradual change in composition between layers, characteristic of gradient systems. The transition from the metal layer to the ceramic layer is achieved through intermediate layers with a smooth change in the concentration of components, which reduces stress and improves adhesion. The coating is characterized by a uniform structure and high-quality distribution of components throughout its thickness, with no signs of porosity or structural damage. Microstructural images of six-layer coatings (Fig. 1c) show a clear layered structure formed by alternating metal (NiCr) and ceramic (Al_2O_3) layers. The boundaries between the layers are clearly distinguishable. However, scratches and signs of interlayer delamination are observed in the structure. The presence of delamination may be due to the occurrence of significant internal stresses during the coating process. These stresses usually arise due to differences in the thermal expansion coefficients between the metal and ceramic layers. During cooling after deposition, such differences can lead to the formation of residual stresses at the interphase boundaries, which reduces the adhesive strength and contributes to local delamination.

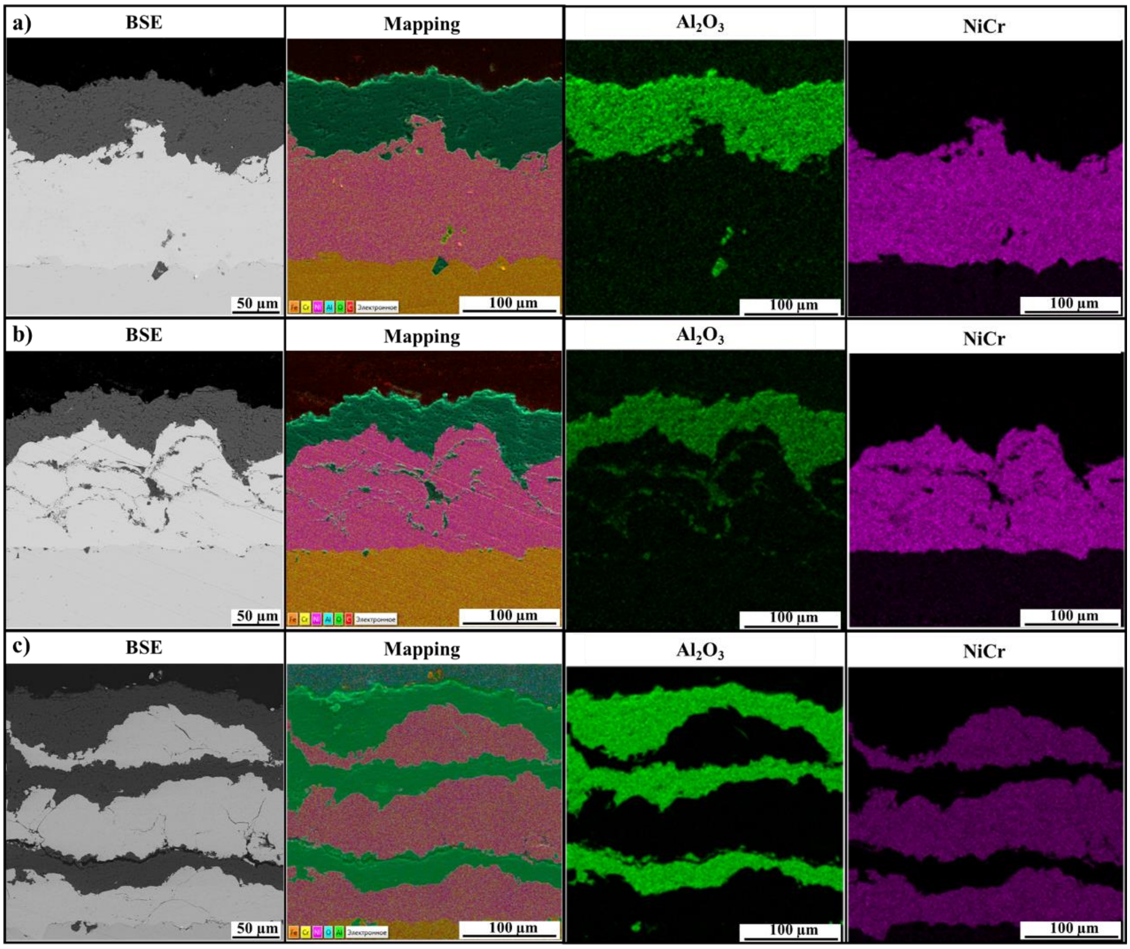

Fig. 1. Microstructure of the cross section of multilayer NiCr/NiCr - Al_2O_3/ Al_2O_3 coatings obtained by detonation spraying: a) three-layer; b) five-layer (gradient); c) six-layer (alternating metal and ceramic layers).

Based on X-ray phase analysis data (Fig. 2), NiCr, α-Al_2O_3, and γ-Al_2O_3 phases were identified in the composition of multilayer metal-ceramic coatings. For three-layer and five-layer coatings, the main phase of the surface layer is Al_2O_3, which indicates the predominance of the ceramic component in the upper layer. In the case of a six-layer coating, the main peaks on the diffractogram correspond to the NiCr phase, which may be due to the fact that the metal layer was closer to the surface. This is explained by the fact that X-rays penetrate to a depth of about 20-30 µm, and with a more complex structure, they capture not only the upper layers but also the inner layers. In addition, the presence of γ-Al_2O_3 phases in some areas of the coatings was observed in all the samples studied-both three-layer and five- and six-layer ones. The appearance of this metastable phase may indicate differences in the crystallization conditions that arise during the deposition process, in particular due to local overheating, uneven temperature distribution across the coating thickness, or changes in the cooling rate.

It should be noted that the thickness and phase composition of the coatings are significantly influenced by the number of detonation pulses (shots) used to obtain each layer. In particular, when obtaining three-layer coatings, each layer was created using 20 detonation shots, which contributed to the formation of layers with increased thickness and, accordingly, ensured a significant total thickness of the entire coating. In the case of five-layer coatings, the number of shots was 10 per layer, and for six-layer coatings, only 5 shots per layer. Thus, as the number of layers increased, the total number of pulses was divided among a larger number of layers, which probably led to a decrease in the individual thickness of each layer. As a result of the decrease in the thickness of the surface layers, especially in six-layer coatings, there is an increased contribution of the inner layers to the X-ray diffraction pattern. This explains the dominance of NiCr phase peaks in the diffractogram of six-layer structures. Nevertheless, it should be emphasized that the Al_2O_3 phase was detected in all types of coatings, including six-layer ones, but its reflection intensity was significantly lower compared to three- and five-layer structures. This indicates the presence of a ceramic component even in the case of thinner top layers, but in smaller quantities, which is confirmed by the reduced intensity of the corresponding diffraction peaks.

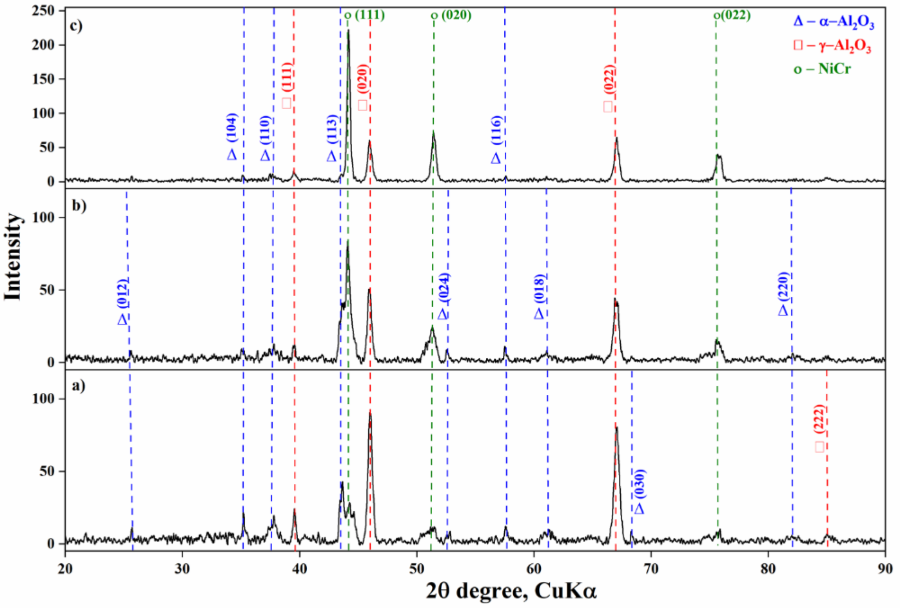

Fig. 2. Diffractogram of multilayer metal-ceramic coatings: a) three-layer coating; b) five-layer coating; c) six-layer coating.

Fig. 3 shows the results of hardness and surface roughness of multilayer metal-ceramic coatings based on NiCr/NiCr - Al_2O_3/Al_2O_3. The data obtained show that the values of these characteristics are within a similar range for all types of coatings studied. That is, despite differences in the number of layers, the behavior of hardness and roughness remains similar for coatings with different layer thicknesses, which indicates the stability of these properties for different structures.

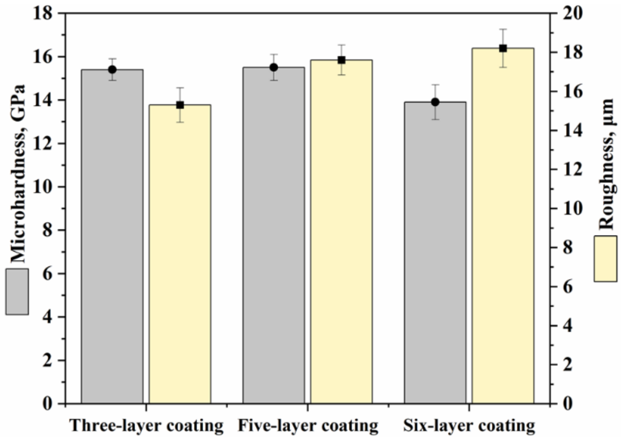

Fig. 3. Hardness and roughness values for multilayer metal-ceramic coatings based on NiCr/NiCr- Al_2O_3/Al_2O_3.

Fig. 4 shows the dependence of the friction coefficient on the distance traveled for three types of multilayer metal-ceramic coatings (three-layer, five-layer, and six-layer) obtained on the basis of NiCr/NiCr-Al_2O_3/ Al_2O_3 at a temperature of 700 °C. According to the results of our previous studies [18], five-layer coatings demonstrated the lowest friction coefficient value-about 0.45 - and were characterized by high wear resistance at room temperature. At the same time, three-layer coatings were characterized by a higher friction coefficient, close to 0.75. For six-layer coatings, a gradual increase in the friction coefficient was observed from an initial value of 0.15 to a stable level of about 0.75. In addition, when testing at room temperature, no significant noise was recorded on the friction coefficient graphs, which indicated the stability of the tribological behavior of the coatings. Analysis of the friction curves obtained in this work confirms that the coating structure and test temperature have a significant effect on its tribological properties.

Fig. 4a shows the dependence of the friction coefficient (red line) on the distance traveled for a three-layer metal-ceramic coating tested at a temperature of 700 °C. In the initial stage of the test, a sharp increase in the friction coefficient is observed, reaching a value of about 0.75, after which it remains at this level for most of the test, showing only slight fluctuations. Insignificant fluctuations in the coefficient may be due to periodic flaking or destruction of the upper ceramic layer of the coating. The green curve shows changes in the depth of penetration of the counterbody into the coating during the test. At the initial stage, a gradual increase in depth is recorded, indicating wear-in and partial destruction of the outer layer. Subsequently, the penetration depth reaches a stable level, indicating stabilization of the wear process. This detailed behavior is characteristic of coatings with a gradient or layered structure, where contact with a harder and more wear-resistant sublayer occurs after the surface layer is removed. The temperature stability reflected by the blue curve confirms the uniform thermal impact on the sample throughout the test, which excludes the influence of thermal fluctuations on the tribological behavior of the coating.

The friction coefficient for the five-layer coating shows more stable behavior compared to the three-layer coating (Fig. 4b). In the initial stage of the test, a smooth increase in the coefficient value to a level of about 0.6 is observed, after which the curve reaches a stable state with insignificant fluctuations. After a distance of about 60 m, a moderate increase in the friction coefficient is recorded, which is probably due to the wear of the top layer and the beginning of interaction with deeper layers of the coating, which have different tribological properties. The penetration depth of the counterbody (green curve) remains at a relatively low and stable level throughout the test, which indicates the high wear resistance of the coating. The temperature regime (blue curve) remains stable throughout the experiment.

For six-layer coatings, the friction coefficient (red curve) increases rapidly at the initial stage of the test and reaches a value of about 0.6. Significant fluctuations are observed in the interval from 20 to 70 m, which may indicate unstable friction associated with the heterogeneity of the upper layers of the coating and their gradual destruction. The peak intensity of the fluctuations is recorded at approximately 60-70 m, after which the curve stabilizes at a level of about 0.6, indicating the establishment of a more uniform mode of interaction with the hardened inner layers. The penetration depth of the counterbody (green curve) shows steady growth in the initial stage, and then reaches a stable level with local irregularities, probably due to the alternation of hard and softer intermediate layers in the coating structure. This indicates gradual wear throughout the entire thickness of the layered coating structure. The temperature regime (blue curve) also remains constant throughout the test.

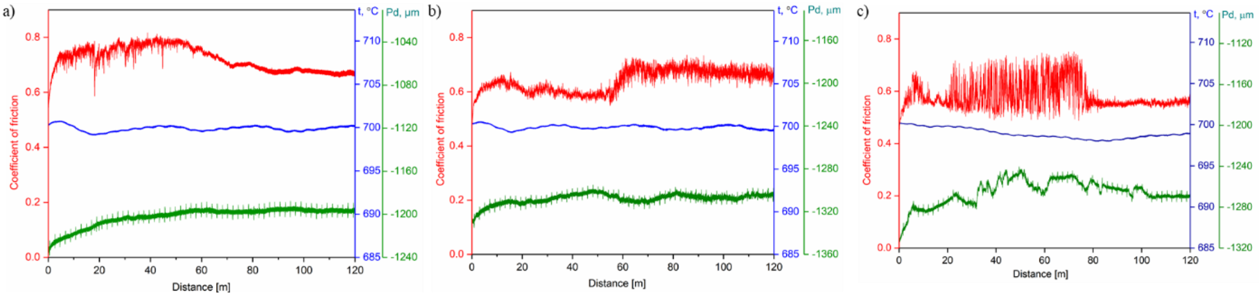

Fig. 4. Dependence of the friction coefficient on distance for multilayer metal-ceramic coatings at a temperature of 700 °C.

4. Conclusion

In this work, multilayer metal-ceramic coatings based on NiCr/NiCr - Al_2O_3/Al_2O_3 were successfully obtained by detonation spraying with a different number of layers - three-layer (NiCr, 50% NiCr - 50% Al_2O_3, Al_2O_3), five-layer (NiCr, 75% NiCr - 25% Al_2O_3, 50% NiCr - 50% Al_2O_3, 25% NiCr - 75% Al_2O_3, Al_2O_3) and six-layer structures (NiCr, Al_2O_3, NiCr, Al_2O_3, NiCr, Al_2O_3). A comprehensive analysis, including scanning electron microscopy, X-ray phase analysis, microhardness and roughness measurements, and tribological tests at 700 °C, allowed us to establish the influence of the coating structure on its performance characteristics.

It was found that the best results were demonstrated by five-layer coatings formed as a gradient structure with a smooth transition from the metal to the ceramic phase. This structure ensured a reduction in internal stresses and increased adhesion between the layers, which was confirmed by microstructural homogeneity, the absence of porosity, and defects at the interphase boundaries. X-ray phase analysis revealed the predominance of the Al_2O_3 ceramic phase in the upper layer of the coating. The microhardness and roughness of the five-layer coatings were comparable to other types of coatings, while demonstrating uniform distribution over the surface. Tribological tests showed that the five-layer coatings had the lowest and most stable coefficient of friction (~0.6) among all the samples studied, as well as the minimum penetration depth of the counterbody, which indicates high wear resistance.

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Conflict of interest

The authors declare no conflict of interest.

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