Hindawi
Advances in Tribology
Volume 2023, Article ID 6684656, 9 pages
https://doi.org/10.1155/2023/6684656

Research Article

Influence of Spraying Parameters on the Structure and Tribological Properties of Cr_3C_2-NiCr Detonation Coatings

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Received 7 February 2023; Revised 4 March 2023; Accepted 15 March 2023; Published 31 March 2023

Academic Editor: Dae-Eun Kim

In this work, the influence of spray parameters on the formation of the microstructure, phase composition, and the tribological properties of detonation flame sprayed coatings was studied. It was determined that the chemical composition of Cr_3C_2-NiCr coatings during detonation spraying depends on the degree of filling the barrel with an explosive gas mixture. The degree of filling the barrel with an explosive gas mixture at 73% leads to a decrease in the content of carbide phases, and at 57% filling of the barrel, an increase in carbide phases is observed. It is established that the decrease of the filling degree leads to the increase of hardness and wear resistance of the Cr_3C_2-NiCr coatings since the hardness and wear resistance of the coating material deposited at 57% is higher than at 65% and 73%; this is due to the increase in the carbide phase Cr_3C_2. Detonation flame sprayed Cr3C2-NiCr gradient coatings have been developed in this study, which is carried out by varying the spray parameters. It was found that in the gradient coating, Cr_3C_2-NiCr carbide phases gradually increase from the depth to the surface. The obtained gradient coating closer to the substrate consists of the $CrNi_3$ phase, while the coating surface consists of $CrNi_3$ and Cr_3C_2 phases.

1. Introduction

Coatings based on Cr_3C_2-NiCr are used to protect the surface of parts operating at high temperatures and in corrosive environments from corrosion and wear [1, 2], which is provided by the ability to bind the solid phase of Cr_3C_2 carbide with the NiCr matrix and to create a high density of the coating material [3, 4]. Various spray technologies are used for coating deposition: atmospheric plasma spraying (APS), high velocity oxygen fuel spraying, air-fuel spraying (HVOF, HVAF), and detonation spraying [5–8]. In the process of coating Cr_3C_2-NiCr powder by thermal methods, a change in the chemical composition of the material is observed due to a decrease in the carbide content in the coating due to elastic rebound [9–15] and due to oxidation [16, 17] caused by the presence of oxygen and superheated water vapor in the combustion products [18, 19]. During thermal spraying of Cr_3C_2-NiCr powder under the influence of high temperatures (several thousand degrees) and a combustion gaseous oxidizing atmosphere, the higher carbides (Cr_3C_2) are depleted from carbon to lower carbides ($Cr_{23}C_6$, Cr_7C_3). Oxidation of carbide particles occurs as well as the migration of dissociation products of carbide particles into the metal matrix [20, 21]. Since the highest carbide Cr_3C_2 has the highest performance characteristics (erosion, corrosion, and wear), the processes of significant decarbonization of carbide particles are undesirable. Various methods of thermal spraying have been proposed for obtaining Cr_3C_2-NiCr coatings with a sufficient amount of higher carbide. One promising method is to obtain coatings with a gradient structure by detonation spraying in which the chemical composition of the coatings changes along with their thickness. Gradient coatings exhibit lower stresses at the substrate-coating interface and can be operated under high mechanical loads [22].

Detonation spraying is widely used to obtain gradient coatings [23–25]. Detonation spraying of gradient coatings is performed by alternating shots from two powder dispensers with different powders [22]. The coating obtained this way has a layered structure, which does not provide high physical and mechanical properties [26]. Therefore, we developed a method of obtaining composite gradient coatings by varying the spray parameters during spraying, i.e., after a certain number of shots, the technological modes (the barrel filling degree, gas ratio, etc.) change [22–25]. To implement this method, it is necessary to apply a detonation setup with a single powder dispenser filled with composite powder.

The purpose of this work is to study the influence of detonation spraying process parameters on the phase composition, hardness, and tribological properties of Cr_3C_2-NiCr coatings and to obtain a gradient Cr_3C_2-NiCr coating by detonation spraying methods.

2. Materials and Methods

2.1. Materials. The ferrite-perlite steel 12Kh1MF (DIN 14MoV63) was chosen as a substrate. This substrate used steel elements of boilers where high wear and corrosion resistance is required. The chemical composition of 12Kh1MF steel is given in Table 1.

Powder Cr_3C_2-NiCr (75/25) (H.C. Starck: AMPERIT® 584.054); powder size of 10–45 μm was used as a spraying material.

2.2. Spraying Methods and Characteristics. A CCDS2000 detonation system (LIH SB RAS, Novosibirsk, Russia) with a system of electromagnetic gas valves regulating the fuel and oxygen supply and controlling the blowing of the system was used to produce the coatings. Details of this system are described in detail in [26]. The general view and a schematic diagram of the detonation spray process are presented in Figure 1. To perform the coating of the barrel which is filled with an explosive gas mixture, a dosed portion of the powder is thrown into the barrel and a detonation is triggered by an electric spark. The detonation products heat the powder particles until they melt and accelerate them with high velocity onto a part mounted in front of the gun barrel. On impact, microwelding occurs and the powder is firmly (on a molecular level) bonded to the surface of the part. After each shot, the barrel is purged with nitrogen to clean the detonation products residue. The desired thickness is achieved by a series of successive shots, during which the object can be moved using a manipulator [27].

The Cr_3C_2-NiCr coating was obtained at different volumes of barrel filling. The gradient Cr_3C_2-NiCr coating was obtained by gradually changing the volume of barrel filling with an explosive gas mixture in the process of detonation spraying, i.e., by gradually reducing the volume of barrel filling with an explosive gas mixture from 73% to 57%. We obtain a gradient structure in which the phase composition changes from the substrate to the surface (the metal phase smoothly transitions to the ceramic phase). Table 2 shows the modes of obtaining coatings based on Cr_3C_2-NiCr.

The phase composition of the obtained coatings based on Cr_3C_2-NiCr was studied using X-ray diffractometer X'PertPRO (Philips Corporation, Amsterdam, the Netherlands) with Cu-Kα-radiation, voltage 40 kV, and current 30 mA. The diffractograms were interpreted using HighScore software, and measurements were made in the 2θ range of 20°–90°, with a step of 0.02 and a counting time of 0.5 s/step. The adhesive strength of the coatings was determined by the detachment method. Coating adhesion strength tests were carried out on an Elcometer 510 instrument. A 10 mm stop was glued with a special 2-part adhesive to the Cr_3C_2-NiCr coating. Then, it was torn off from the substrate using an automatic adhesion meter at a rate of 0.80 MPa/s. Microimages of the coating surface were obtained using a light microscope (model Altami MET 5S, LLC "Altami," Russia, St. Petersburg). The surface roughness of the coatings Ra was evaluated using a profilometer model 130 (JSC "Plant PROTON," Moscow, Russia). Microhardness of the coating was measured in the cross section in accordance with GOST 9450-76 (ASTM E384-11) on microtestometer Metolab 502 (Metolab, Russia), with loads 1 N and an exposure time of 10 seconds. Tribological tests for sliding friction were carried out on a tribometer TRB^3 (Anton Paar Srl, Peseux, Switzerland) using the standard "ball on disc" technique (international standards ASTM G 133-95 [28] and ASTM G99 [29]), where a ball made of SiC-coated steel, with a diameter of 6.0 mm and under a load of 6 N, is being used as the opposing body. The system is moving at a constant linear speed of 5 cm/s, and the radius of curvature of the wear is 5 mm. The total distance covered by the system due to friction is 200 m. The surface morphology was investigated by scanning electron microscopy (SEM) using backscattered electrons (BSE) on a JSM-6390LV scanning electron microscope (Joel, Tokyo, Japan).

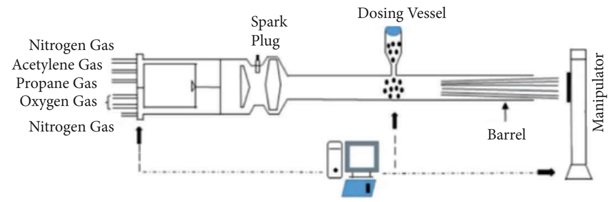
3. Results and Discussion

Figure 2 shows the diffractograms of the obtained coatings at different barrel fills. The following phase components were detected in the coatings: Cr_3C_2, Cr_7C_3, $Cr_{23}C_6$, Cr_2 O, and $CrNi_3$ phases. From the diffractograms, we can see that the phase composition of the coatings changes depending on the degree of barrel filling. With the degrees of barrel filling at 57% and 65%, the diffractograms show the peaks of carbide and oxide phases at 64.3° (Cr_3C_2 and Cr_2O) and at 82.4° (Cr_3C_2, Cr_7C_3, $Cr_{23}C_6$). The combination of solid phases of chromium oxide and chromium carbide significantly increases the durability of the resulting material in wear conditions. And at 57% of the barrel filling, there is an increase in the intensity of carbide phases. With the volume of the barrel filling at 73%, a new $CrNi_3$ peak and an increase in the intensity of the $CrNi_3$ phase peaks are observed in the diffractograms. This is due to the high temperature of the powder during spraying. When the barrel is 73% full, the temperature in the reaction zone can reach 3500°C. The structure of the $CrNi_3$ phase is more elastic relative to others and provides its high adhesion to the substrate [30].

Table 1: Chemical composition (%) of 12Kh1MF steel.

C	Ti	Mn	Ni	S	P	Cr	Mo	V	Cu	Fe
0.1–0.15	0.17–0.37	0.4–0.7	to 0.3	to 0.025	to 0.03	0.9–1.2	0.25–0.35	0.15–0.3	to 0.02	96,5


(a)



(b)

Figure 1: Computerized detonation complex SSDS2000: (a) general view and (b) schematic diagram of the setup.

Table 2: Modes of coatings based on Cr_3C_2-NiCr.

Samples	Volume of barrel filling (%)	Number of shots	Spraying distance (mm)
No. 1	57	30	150
No. 2	65	30	150
No. 3	73	30	150
No. 4	57-65-73	30 (10-10-10)	150

Figure 3 shows the results of the adhesion strength of the coating. Coatings obtained by filling the volume of the barrel with 73% have a high adhesive strength compared to the rest. This is due to an increase in the proportion of the $CrNi_3$ phase (confirms the results of X-ray structural analysis), which is more elastic and ensures its high adhesion to the substrate.

Figure 4 shows the surface micrographs and the results of measuring the roughness of the obtained coatings based on Cr_3C_2-NiCr obtained at different volumes of filling the barrel with an explosive gas mixture. The surface of all coatings has an inhomogeneous structure with small pores, which is a characteristic of detonation coatings. The Ra parameter, which is the arithmetic mean deviation of the profile, was chosen as the main coating roughness parameter. The roughness parameter of coatings obtained at barrel fills up to 57%, 65%, and 73% has similar values in the range of Ra = 5.61–6.32. This may be due to the difference in the impact of the shock wave, which leads to the compaction of the coating. The roughness test results showed that at different barrel fill volumes, the roughness of the spray coatings is not significantly affected.

Measurement of microhardness of the samples was carried out according to GOST 9450-76 (ASTM E384-11), at loads on an indenter $P = 1$ N and exposure time 10 s. Figure5 shows the results of hardness measurement. According to the results of the measurements, it can be stated that the coating obtained at 57% of filling has high hardness (11.9 GPa) in comparison with the coatings obtained at other modes, which is also apparently due to the increase in the intensity of the carbide phase Cr_3C_2. Of the three samples, the coating obtained at 73% filling has a low hardness, and this is due to the increase in the CrNi3 phase, which is more ductile(confirms the results of X-ray structural analysis).

Tribological parameters were evaluated by the method of sliding friction on a tribometer, where data on the friction coefficient of the surface of the obtained coatings were obtained. According to the graphical data (Figure 6), the coatings obtained at different fillings of the barrel have a coefficient of friction in the range of 0.2–0.5. Based on the data obtained, graphs were built, as well as using a special program, we obtained the values to calculate the amount of wear at different volumes of barrel filling (Figure 6(b)). As can be seen from Figure 6(b), at 57% filling of the barrel with explosive gas mixture, the obtained coating has a low volume of wear ($0.0132\,mm^3$) and a low coefficient of friction (0.263), which suggests an increase in wear resistance, which is most likely due to the increased proportion of the Cr_3C_2

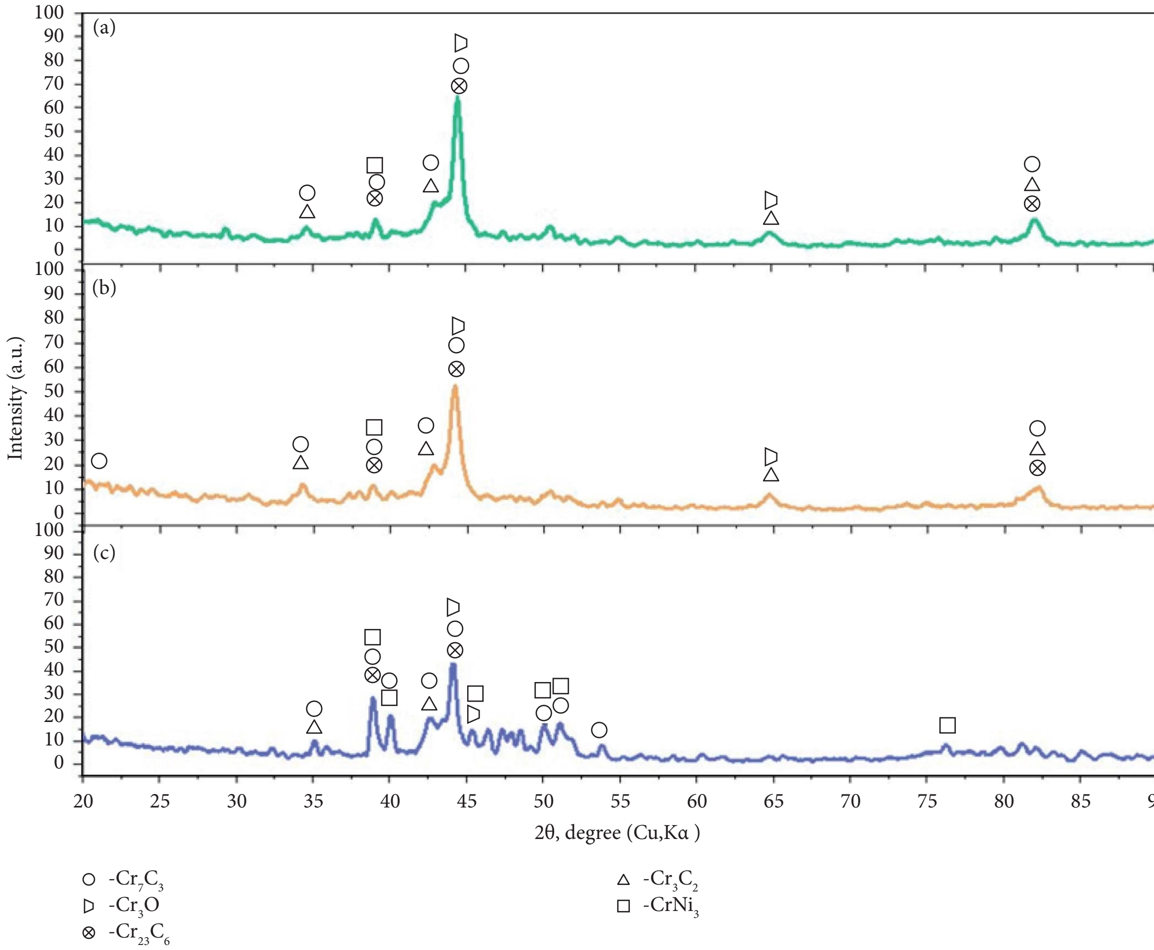


Figure 2: Surface diffractograms of the Cr_3C_2-NiCr coating material at different barrel fills: (a) 57%; (b) 65%; and (c) 73%.

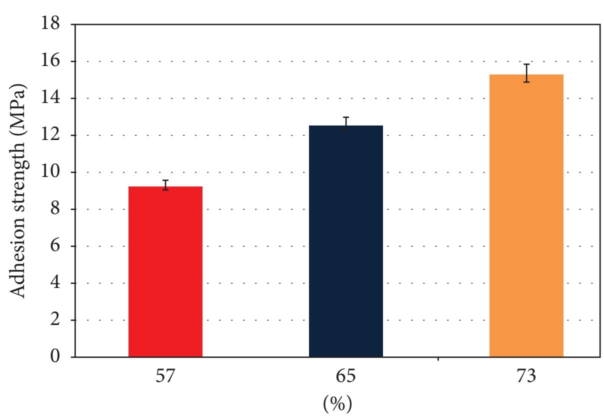


Figure 3: Graph of the adhesion strength of the Cr_3C_2-NiCr coating material at different barrel fills: (a) 57%; (b) 65%; and (c) 73%.

carbide phase, which has high resistance to the wear material. The coefficient of friction of the coating obtained at 73% of barrel filling with the explosive gas mixture is 0.427 and it has a high wear volume of 0.0227 mm^3. This is connected with a lower share of the carbide phase Cr_3C_2 and an increase of the phase $CrNi_3$ which has a lower wear resistance than Cr_3C_2 (confirms the results of X-ray structural analysis). Table 3 shows the results of the tribological testing of Cr_3C_2-NiCr coatings.

Based on the study of the influence of the spraying mode on the structure and properties of Cr_3C_2-NiCr -based coatings, we have developed a method of obtaining gradient coatings. Prerequisites for the development of obtaining gradient coatings by the method of detonation spraying are our previous works [18, 20]. This method is carried out on a detonation flame spraying system with one powder dispenser using a composite powder based on Cr_3C_2-NiCr by changing the spray parameters during the spraying process. Furthermore, the structural-phase state and properties of the gradient coating based on Cr_3C_2-NiCr were studied, which was obtained by gradually reducing the volume of the barrel filling from 73% to 57% with a gas mixture. The choice of such spraying parameters is based on the abovementioned experimental results and is aimed at obtaining coatings in which the distribution of carbide phases in the Cr_3C_2-NiCr gradient coating gradually increases from the substrate to the surface, and accordingly, a large number of carbide phases are formed on the coating surface uniformly increasing from the substrate to the coating surface. It is assumed that the Cr_3C_2-based carbide that forms on the surface when the barrel is filled with 57% explosive gas mixture contributes to wear resistance and hardness, while

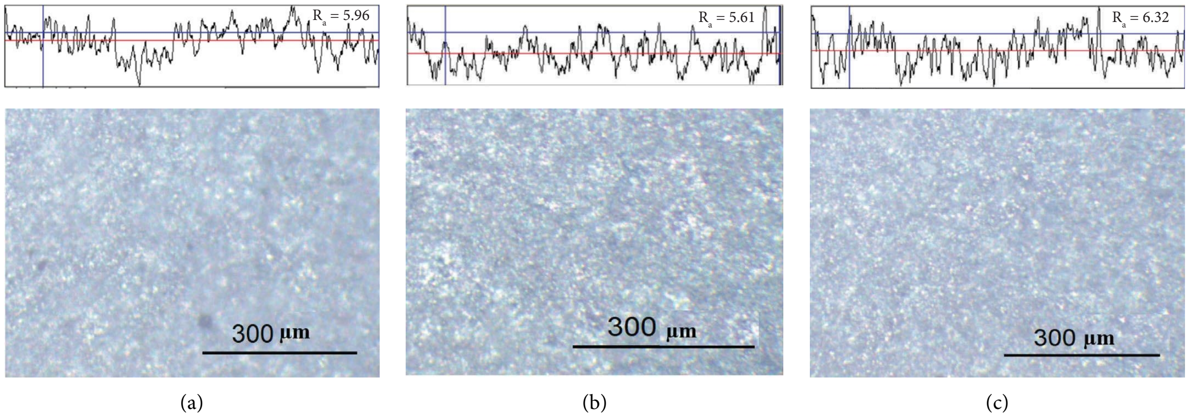


FIGURE 4: Microphotographs and surface roughness profile of Cr_3C_2-NiCr coatings: the barrel filling volume to (a) 57%; (b) 65%; and (c) 73%.

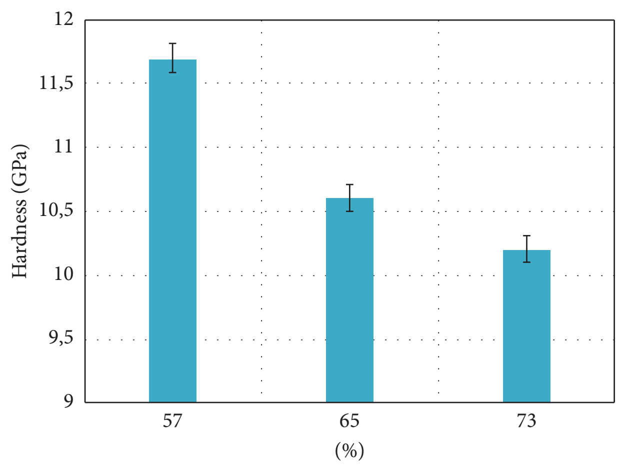


FIGURE 5: Graph of the hardness distribution of the Cr_3C_2-NiCr-based gradient detonation coating as a function of barrel filling.

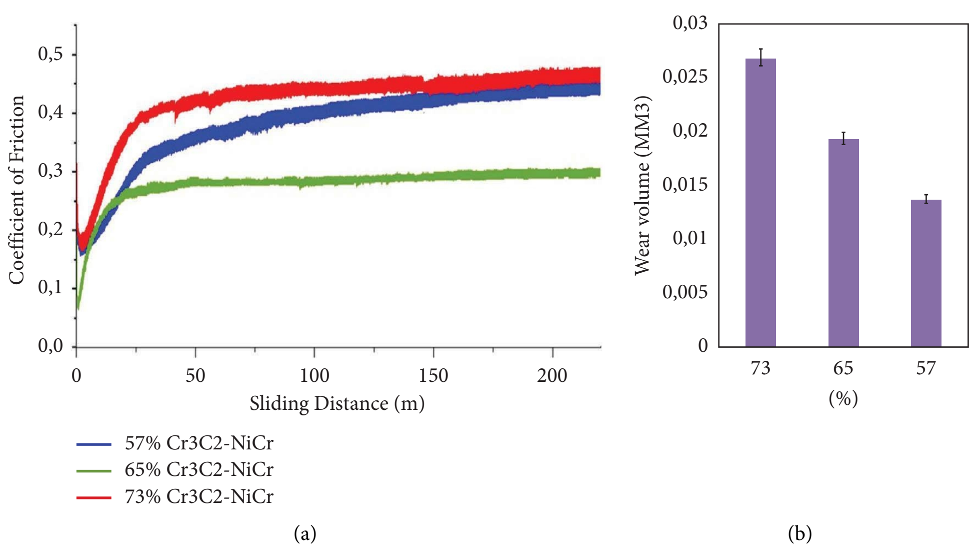


FIGURE 6: Results of tribological testing of Cr_3C_2-NiCr coatings according to the "ball-disk" scheme. (a) Coefficient of friction and (b) wear volume.

Table 3: Results of the tribological testing of Cr_3C_2-NiCr coatings.

Samples	Volume of barrel filling (%)	Coefficient of friction	Wear volume (mm^3)
No. 1	57	0.263	0.0132
No. 2	65	0.366	0.0196
No. 3	73	0.428	0.0227

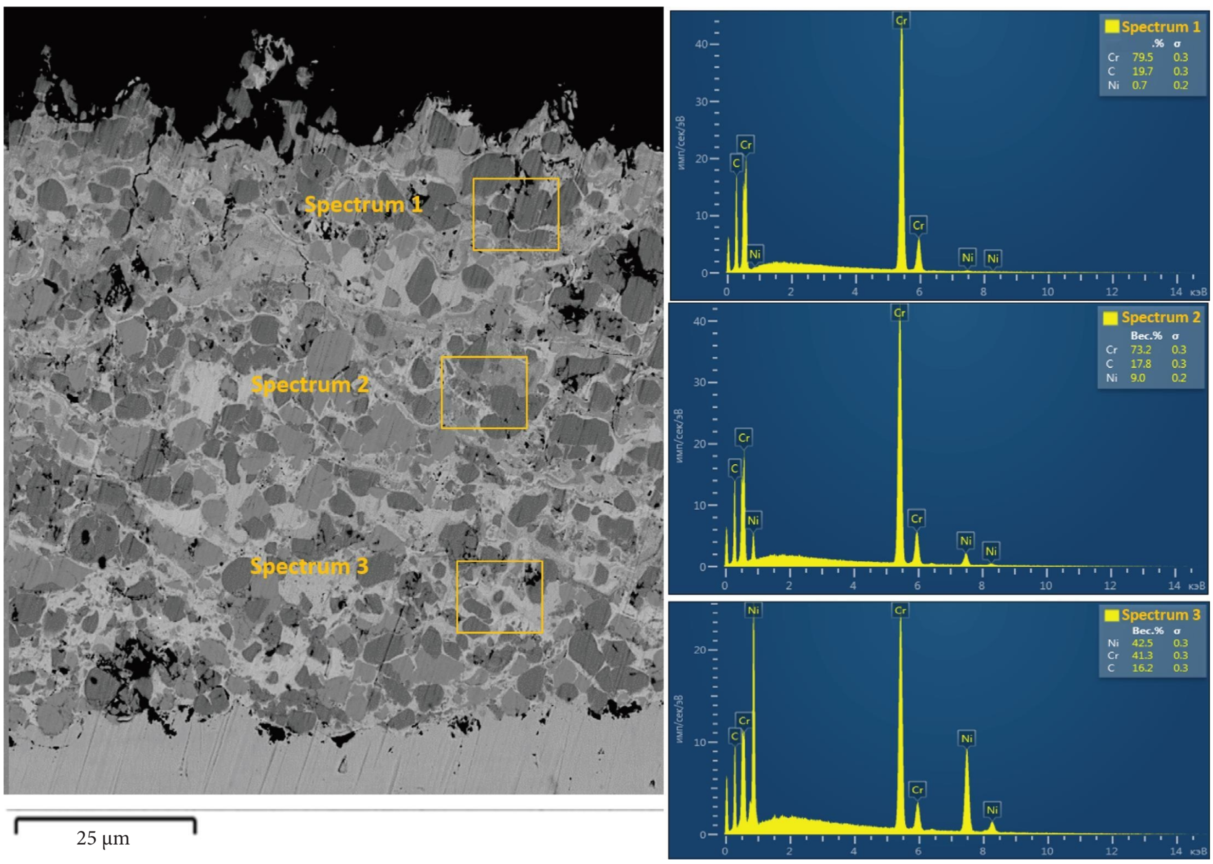


Figure 7: SEM images of Cr_3C_2 gradient coating and results of EDS analysis performed on three points.

the $CrNi_3$ phase formed close to the substrate provides a high adhesion strength of the coatings.

Figure 7 shows SEM images of the Cr3C2-NiCr gradient coating and the results of EDS analysis performed on three points. According to the results of EDS analysis, we can see the increase of the Cr, C content from spectrum 3 to spectrum 1. It is also established that on the surface of gradient coatings, layers with a high content of Cr, C are formed, which is confirmed by elemental analysis.

According to the results of SEM imaging (Figure 8) and EDS analysis along the lines of the cross section of the sample, we can see that the coating has a gradient structure, Cr having a dark shade, which gradually increases from the substrate to the coating surface, and Ni decreases from the substrate to the surface of the substrate. EDS analysis along the lines was carried out in the direction from the surface to the substrate.

Figure 9 shows a graph of the microhardness distribution over the depth of the gradient coating based on Cr_3C_2-NiCr

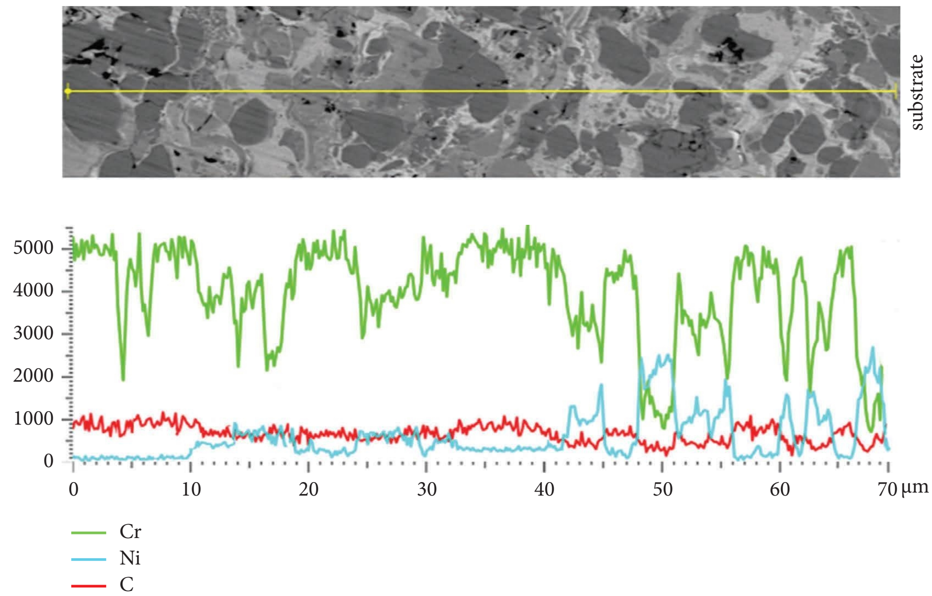


Figure 8: SEM images of the cross section and element depth distribution of the gradient coating Cr_3C_2-NiCr.

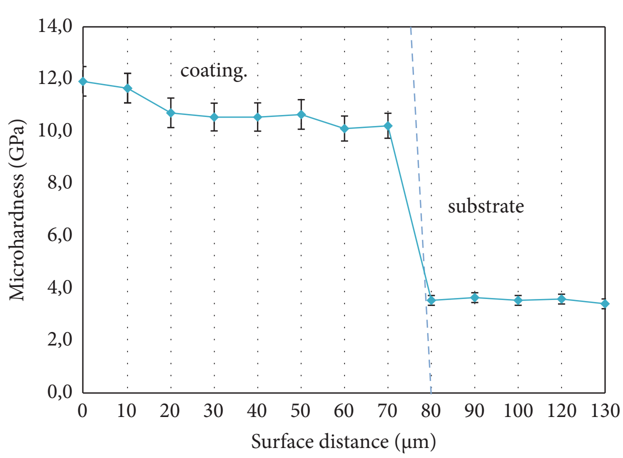


Figure 9: Graph of the depth hardness distribution of the gradient coating (57-65-73) based on Cr_3C_2-NiCr obtained by detonation spraying.

obtained by detonation spraying. The graph of dependence of microhardness on the depth of the gradient coating Cr_3C_2-NiCr demonstrates a smooth decrease of hardness from the surface to the substrate. At the coating surface, the hardness has a value 11.9 GPa and closer to a substrate has a value 10.1 GPa.

4. Conclusion

According to the evaluation and analysis of all obtained results, we can draw the following main conclusions on the present research work:

(i) It was found that spraying at different barrel fill volumes had no effect on the roughness of the sprayed coatings;

(ii) It has been established that detonation coating technologies provide, at optimal modes, the formation of wear-resistant coatings from the metal-ceramic material of the Cr_3C_2-NiCr system;

(iii) It has been determined that *t* different filling degrees of the combustion chamber of detonation spraying of coatings provide increase of strength properties and hardness of matrix without significant degradation of primary carbide particles and also increase in quantity of carbide particles and creation of chrome oxides in the near-surface layer;

(iv) A method of obtaining gradient coatings based on Cr_3C_2-NiCr using a detonation setup, with one powder dispenser, has been developed. This method is carried out using one type of composite powder on the basis of Cr_3C_2-NiCr by changing technological parameters during the spraying process;

(v) The study of the elemental composition by the SEM method (EDS) showed that the detonation coatings based on Cr_3C_2-NiCr have a gradient structure because there is a gradual increase in the concentration of elements Cr, C from the substrate to the coating surface as well as a decrease in the concentration of the element Ni from the surface to the substrate;

(vi) Gradient coating hardness test results showed a smooth hardness change from surface to substrate.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

This research has been funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant no. AP09261164).

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