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Effect of HVOF spraying parameters on the structural-phase composition and mechanical properties of ZrCN coating

The article presents the results of a study on the influence of HVOF spraying parameters on the phase composition, mechanical properties, and adhesion characteristics of zirconium carbonitride (ZrCN) coatings. X-ray diffraction analysis of the ZrCN coatings revealed the presence of ZrCN, ZrC, ZrN, ZrO, Fe, and FeN phases, indicating a complex coating structure and possible oxidation and elemental diffusion processes. The formation of ZrC and ZrN is attributed to the thermal decomposition of ZrCN powder during the coating process, while the presence of the oxide phase ZrO is explained by the use of an oxygen-containing gas mixture during HVOF spraying. The microhardness of the ZrCN coatings reaches values in the range of 1500–1800 HV, depending on the spraying parameters. Adhesion test results showed that the maximum coating bond strength under tensile load was 7.49 MPa. Optimal coating characteristics were achieved at the following spraying parameters: substrate distance of 35–40 cm, propane pressure of 1.7 bar, air pressure of 2.6 bar, and oxygen pressure of 2.8 bar. These conditions allow the formation of a dense, wear-resistant coating structure with enhanced performance characteristics.

Keywords: HVOF, microhardness, adhesion, structure, phase composition, ZrCN coating, X-ray diffraction

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Introduction

Currently, ensuring high wear resistance and durability of cutting tools of technological equipment is one of the key tasks in mechanical engineering and metalworking. Cutting tools and parts operating under conditions of intensive wear and high mechanical loads require the use of wear-resistant protective coatings [1]. Both physical and chemical deposition methods are actively used to form protective and functional coatings. Physical processing methods include physical vapor deposition, plasma spraying, and magnetron sputtering, which provide dense, wear-resistant coatings with high adhesion to the substrate [2–6]. Additionally, to increase wear resistance and corrosion resistance of materials, sol-gel technologies and laser coating methods are actively used, ensuring control over the composition and structure of the formed layers [7, 8].

Among the above methods, the most promising is the HVOF technology, which allows obtaining nanostructured coatings. HVOF technology has become widespread in many industries due to its flexibility and cost-effectiveness in obtaining high-quality coatings [9–11]. The physical and mechanical properties of HVOF sprayed coatings largely depend on the nano- or microstructure of the coating, which in turn largely depends on the physical and chemical state of the particles at the point of impact on the substrate, such as speed, temperature, degree of melting and oxidizer content [12–14]. In particular, coatings that can withstand wear, high mechanical loads and chemically aggressive influences are of particular importance. In this regard, ZrCN coatings demonstrate excellent characteristics under such conditions [15, 16]. It has been proven that ZrCN-based ceramics have high thermal stability and resistance to physical and chemical environments, which makes them an excellent candidate for protective coatings of cutting tools and metal parts operating in extreme conditions [17–19]. It was found that the grains of ZrN/ZrCN coatings were denser, finer and more compact than those of Zr/ZrN coatings. Accordingly, higher values of hardness, modulus and H/E were demonstrated by ZrN/ZrCN coatings [20].

The aim of this study is to investigate the influence of HVOF spraying mode parameters on the phase composition and mechanical properties of ZrCN coating.

Materials and methods

The ZrCN coatings were deposited using the HVOF method on a Termika-3 system [21], which is equipped with a control panel that allows precise adjustment of the gas supply pressures. Figure 1 presents both a visual and schematic illustration of the equipment used. As the pressure increases, the gas components are mixed inside the chamber, after which the powder material with a metered feed enters the burner, where it is transported by compressed air supplied by the compressor.

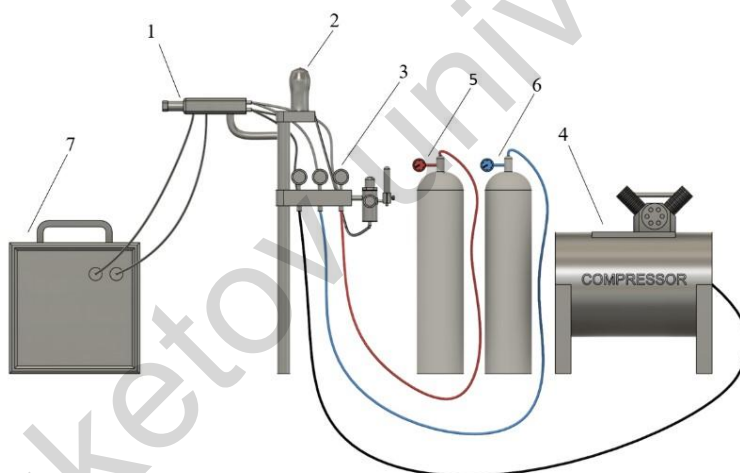


Figure 1. External view and structural diagram of the HVOF spraying system: 1—torch; 2— powder feeder; 3—gas control panel; 4—compressor; 5—gas cylinder (C_3H_8); 6—gas cylinder (O_2); and 7—chiller

At the outlet of the burner, the powder particles enter the flame zone, where they are heated to a state that ensures their plastic deformation. Then the molten particles are directed to the pre-prepared surface of the substrate, forming a uniform protective coating. The ZrCN powder with a particle size range of 20–100 μm exhibits an irregular polyhedral fragmented morphology, which is known to have lower flowability compared to spherical particles. The main spraying parameters are given in Table 1. Steel grade 65G [22], which belongs to the group of high-carbon, alloyed steels, was used as a substrate.

Table 1

Coating application parameters

№	Distance	Propane	Air Pressure	Oxygen	Powder
Sample a	35–40 cm	1.7 bar	2.6 bar	2.8 bar	ZrCN
Sample b			2.8 bar		
Sample c			3 bar		

Table 2 shows the chemical composition of 65G steel. Before spraying, the substrate surface was mechanically processed (grinded) to remove oxide films, and then sandblasted to improve coating adhesion.

Table 2

Chemical composition of steel 65G [22]

Steelgrade	Mass fraction of elements, %			
	Carbon	Silicon	Manganese	Chromium
65G	0.62–0.70	0.17–0.37	0.90–1.20	0.25

The phase composition of the obtained ZrCN-based coatings was studied using an X'Pert PRO X-ray diffractometer with Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$), a voltage of 40 kV and a current of 30 mA. The scanning angle range was from 20.01° to 89.99° with a step of 0.02° and a data accumulation time of 2 s. The diffraction patterns were processed using High Score Plus software.

Microhardness was investigated using the Vickers method in accordance with GOST 9450-76 on the HLV-1DT microhardness tester. A diamond tetrahedral pyramid with angles of 136° was used as an indenter in the study. During the measurement, a load of $HV_{0.5}$ was applied to the surface of the sample, and the indenter was held for 10 seconds. Then the diagonal dimensions of the input traces (d1 and d2) were determined with accuracy [24].

To study the adhesive properties of the coatings, tests were carried out in accordance with ASTM D4541-22. Using an Elcometer 510 hydraulic adhesion meter (Elcometer Instruments, Manchester, UK). The strength of the adhesive coatings was determined under the following conditions: hold time 0.50 s; target speed 1.00 MPa/s; backing size 20 mm.

Results and discussion

To study the phase composition of the ZrCN powder intended for spraying, X-ray phase analysis was carried out, the diffraction pattern of which is shown in Figure 2. It can be seen from the figure that the presence of the ZrCN peak next to the ZrC, ZrN peaks indicates its partial preservation [23] and confirms its decomposition with the formation of zirconium nitride. Zirconium carbonitride combines high hardness with good plasticity, which contributes to the increased resistance of the material to the formation and propagation of cracks.

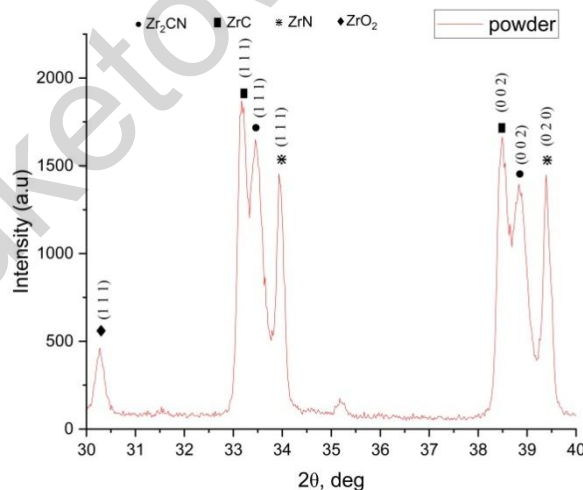


Figure 2. X-ray diffraction pattern of ZrCN powder

X-ray diffraction patterns of three coating samples are shown in Figure 3. The study of the phase composition of the sprayed coatings by the HVOF method showed the presence of the following phases: ZrCN, ZrC, ZrN, ZrO, Fe and FeN. The X-ray diffraction results confirmed that $ZrC_{1-x}N_x$ crystallizes in a face-centered cubic (FCC) structure, and its diffraction peak is located between the control peaks of ZrC and ZrN, which indicates complete mutual solubility of these phases [24], the parameters of the ZrCN crystal lattice are cubic system, space group Fm-3m. Compared to titanium and hafnium carbonitrides, ZrCN has greater plasticity, which has a positive effect on the crack resistance of ceramics made on its basis [25]. In

works [26, 27], high values of hardness and critical intensity factors were revealed. In addition to high hardness, an important advantage of the coating is its relatively high thermal conductivity, which reduces the risk of thermal damage [28]. The formation of ZrC and ZrN phases is associated with thermal decomposition of ZrCN powder. Addition of large amounts of oxygen to the ZrN structure can distort its crystal structure, introduce defects and promote the formation of an amorphous structure. A decrease in grain size and an increase in the lattice constant are associated with the presence of oxygen in the coatings. These effects will manifest themselves as a broadening of peaks in X-ray diffraction patterns [29]. Many nitrides such as ZrN crystallize in sodium chloride type structures, and in an ideal perfect crystal first order Raman scattering is forbidden. However, it is known that deposited coatings contain vacancies which cause distortion of the structure [30], and as a consequence the Raman spectrum consists of broadened bands due to disorder and second order processes.

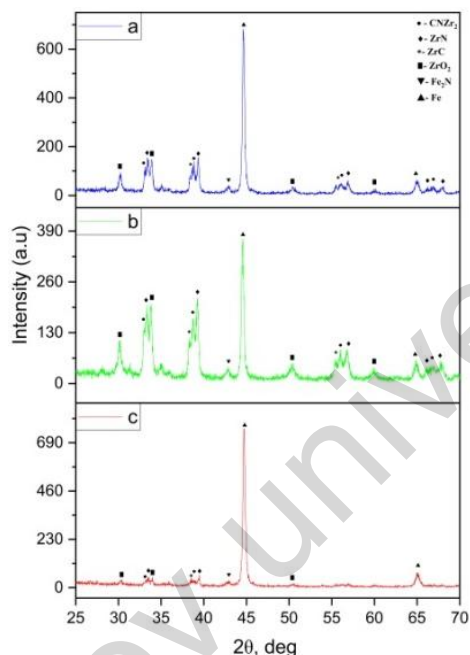


Figure 3. X-ray diffraction pattern after ZrCN coating HVOF modes:
 sample *a* distance 35–40 cm, fuel pressure 1.7 bar, air pressure 2.6 bar, oxygen pressure 2.8 bar;
 sample *b* 35–40 cm, fuel pressure 1.7 bar, air pressure 2.8 bar, oxygen pressure 2.8 bar
 and sample *c* 35–40 cm, fuel pressure 1.7 bar, air pressure 3 bar, oxygen pressure 2.8 bar

It is known [31] that zirconium oxide can be found in mainly different phases: tetragonal ZrO₂, monoclinic ZrO₂ and cubic ZrO₂. Usually pure t-ZrO₂ in the tetragonal phase exists if Y₂O₃ yttrium oxide is present in the coatings [32]. HVOF promotes the formation of a mixture of c-ZrO₂ and t-ZrO₂, but if the cooling is very fast, more of the cubic phase remains. The formation of the ZrO phase, which possesses a similar cubic lattice structure with the space group Fm-3m, is attributed to the use of an oxygen–propane mixture as the oxidizing environment during high-velocity oxy-fuel spraying. This led to an active interaction of ZrCN with oxygen and a partial loss of carbon. Excess carbon released during the decomposition of ZrCN diffused into the metal matrix, promoting the formation of carbide (ZrC) and oxide (ZrO) phases. All samples exhibit low Zr/(C+N) ratio, $0.3 \leq \text{Zr}/(\text{C}+\text{N}) \leq 0.6$, which suggests the presence of an additional amorphous phase, most likely amorphous C or CN_x. Indeed, these phases were also observed in the TiCN system [33]. The Fe phase was detected at an angle of 44.62°, its main parameters are: cubic system, space group—Im-3m. In addition, the FeN phase, space group P-3m1, with a cubic lattice was identified. At the same time, the main lines of this phase coincide in positions with the shifted lines of the (ZrC)(ZrN) type phases with cubic lattice parameters mentioned earlier. The peaks of the phase lines in sample 3 at angles of 38.76° have characteristic broadenings, indicating the presence of phases of transformed compositions. The microhardness of the coatings and the substrate is an important parameter determining their mechanical properties and performance characteristics. Figure 4 shows the obtained microhardness values for the substrate and ZrCN coatings. The initial microhardness values of the substrate are 400–500 HV, which corresponds to the typical characteris-

tics of hardened steel 65G. This indicates a relatively low hardness of the base material compared to the applied coatings and emphasizes the need to use protective layers to improve wear resistance.

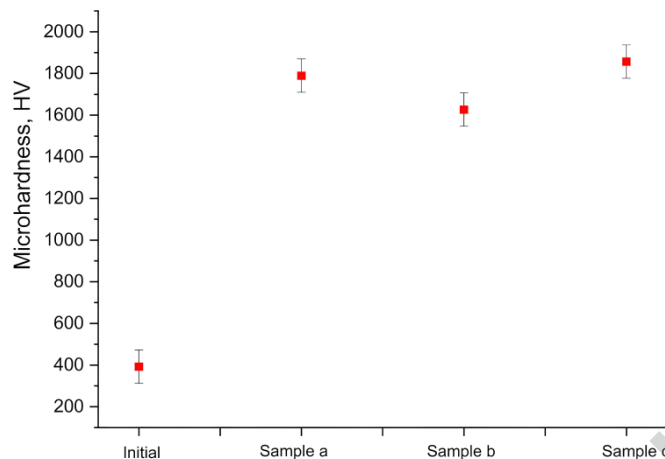


Figure 4. Microhardness values of 65G steel. HVOF modes:
 sample *a* distance 35–40 cm, fuel pressure 1.7 bar, air pressure 2.6 bar, oxygen pressure 2.8 bar;
 sample *b* 35–40 cm, fuel pressure 1.7 bar, air pressure 2.8 bar, oxygen pressure 2.8 bar
 and sample *c* 35–40 cm, fuel pressure 1.7 bar, air pressure 3 bar, oxygen pressure 2.8 bar

After deposition of ZrCN coatings, a significant increase in microhardness is observed, with values varying depending on the sample. Sample *a* shows an increase in microhardness to 1600 HV, indicating the formation of a dense coating with a good degree of particle compaction. Sample *b* reaches a maximum hardness of 1800 HV, which can be associated with the optimal particle size of the coating, uniform phase distribution and minimal porosity. Sample *c* shows microhardness in the range of 1500–1600 HV, which is slightly lower than the second sample, but still indicates high coating strength. A smooth change in microhardness is observed at the coating-base interface, indicating possible diffusion of alloying elements and temperature effects during the spraying process.

Coating adhesion tests were carried out using the pull-off method in accordance with GOST 32299-2013 (ISO 4624: 2002) at a temperature of 20 ± 5 °C no earlier than three days after coating application. To improve the adhesive bond, the coating surface at the gluing point of the “mushroom” was treated with sandpaper, provided and degreased with ethyl alcohol. The adhesive was applied according to the manufacturer’s instructions. Epoxy Adhesive 2214 was applied in an even layer to the surface of the “mushroom”, then the “mushroom” was pressed against the coating and kept until the adhesive hardened, ensuring the centering of the surfaces to be glued. If necessary, excess glue was removed. Using a cutting tool (annular cutter), the coating was cut to the metal around the “mushroom”. Saw cuts were made across the entire coating thickness until the metal appeared, with the cut width being at least 1 mm. Tests were conducted at least 24 hours after gluing the “mushrooms”. To measure adhesion, the “mushroom” was placed in a special adhesion meter device. The adhesion meter’s stop mechanism was hooked onto the “mushroom” and by pressing the handle, a normal tear-off force was applied, the value of which was recorded on the device scale. It should be noted that non-compliance of the coating with the operating conditions of its application (e.g., climatic conditions), insufficient quality of surface preparation and other violations of the application technology lead not only to a decrease in its efficiency and reliability, but also to defective coating.

In Figure 5, the final result was a maximum bond strength at break of 7.49 MPa per square centimeter. But even with such a break, the surface of the sample was not damaged. In sample *b*, we can see distinct places that were attached to the “mushrooms” and it is clear that the surface of the sample was not deformed during the break. When breaking, it was obvious that we only tore off the adhesive, not the surface of the sample. With such indicators, the surface of the sample was not affected in any way and no visual deformations were visible.

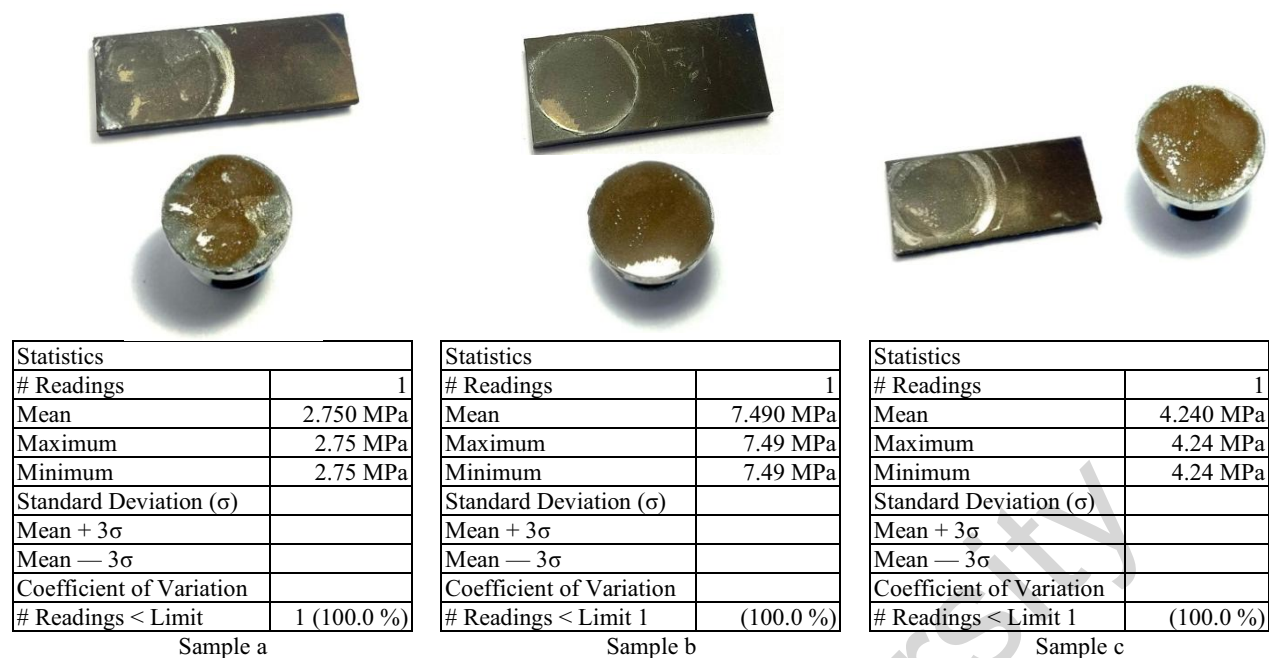


Figure 5. Image of the sample surface and the results obtained after the adhesion test

Conclusion

This research paper presents the results of structural-phase, microhardness and adhesion of ZrCN coatings applied by the HVOF method on 65G steel. The study showed that ZrCN coatings applied by the HVOF method form a complex phase structure including ZrCN, ZrC, ZrN, ZrO phases. The best formation of ZrCN and ZrC phases is achieved at moderate values of atmospheric pressure, which minimizes oxidation and increases the coating density. Sample (b) demonstrates the most favorable phase composition, which confirms its best mechanical properties, such as high hardness, wear resistance and chemical resistance. ZrCN-based coatings have significantly higher microhardness compared to the substrate, which is largely due to the formation of ZrC, ZrN. Sample (b) demonstrates the greatest strength, having a microhardness of 1800 HV, which makes it the most suitable for use under high loads. In addition, the maximum adhesion at adhesion failure is 7.49 MPa per square centimeter, which shows that sample (b) of the coating has high adhesion and has a layered structure characteristic of thermal spraying. The results obtained confirm that HVOF spraying can form coatings with high wear resistance and heat resistance, ensuring reliable operation under extreme conditions.

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HVOF бүрку параметрлерінің ZrCN жабындарының құрылымдық-фазалық құрамына және механикалық қасиеттеріне әсері

Мақалада HVOF бүрку параметрлерінің цирконий карбонитрид (ZrCN) жабынының фазалық құрамына, механикалық қасиеттеріне және адгезиялық сипаттамаларына әсерін зерттеу нәтижелері берілген. ZrCN жабындарының рентгендік дифракциялық талдауы ZrCN, ZrC, ZrN, ZrO, Fe және FeN фазаларының болуын анықтады, бұл күрделі жабын құрылымын және элементтердің ықтимал тотығу және диффузиялық процестерін көрсетеді. ZrC және ZrN түзілуі жабу процесінде ZrCN ұнтағының термиялық ыдырауымен байланысты, ал ZrO оксид фазасының пайда болуы HVOF шашу кезінде оттегі қоспасы қолданылғанымен түсіндіріледі. ZrCN жабындарының микроқаттылығы бүрку параметрлеріне байланысты 1500–1800 HV диапазонындағы мәндерге жетеді. Адгезия сынауларының нәтижелері үзілу кезінде жабынның максималды байланыс беріктігі 7,49 МПа болғанын көрсетті. Оңтайлы сипаттамаларға келесі бүрку параметрлері арқылы қол жеткізілді: субстратқа дейінгі қашықтық 35–40 см, пропан қысымы 1,7 бар, ауа — 2,6 бар, оттегі 2,8 бар. Белгіленген шарттар өнімділік қасиеттері жақсартылған тығыз, тозуға төзімді жабын құрылымын қалыптастыруға мүмкіндік береді.

Кілт сөздер: HVOF, микроқаттылық, адгезия, құрылым, фазалық құрамы, ZrCN жабындары, рентгендік дифракция

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Влияние параметров напыления HVOF на структурно-фазовый состав и механические свойства покрытий ZrCN

В статье представлены результаты исследования влияния параметров высокоскоростного газопламенного напыления (HVOF) на фазовый состав, механические свойства и адгезионные характеристики покрытия карбонитрида циркония (ZrCN). Рентгеноструктурный анализ покрытий ZrCN выявил присутствие ZrCN, ZrC, ZrN, ZrO, Fe и FeN фаз, что свидетельствует о сложной структуре покрытия и возможных процессах окисления и диффузии элементов. Формирование ZrC и ZrN связано с термическим разложением порошка ZrCN в процессе получения покрытия, а образование оксидной фазы ZrO объясняется тем, что при HVOF напылении использовалась смесь кислорода. Микротвердость ZrCN покрытий достигает значений в диапазоне 1500–1800 HV в зависимости от параметров напыления. Результаты испытаний на адгезию показали, что максимальная прочность сцепления покрытия при разрыве составила 7,49 МПа. Оптимальные характеристики достигнуты при параметрах напыления: расстояние до подложки 35–40 см, давление пропана 1,7 бар, воздуха — 2,6 бар, кислорода 2,8 бар. Установленные условия позволяют сформировать плотную, износостойкую структуру покрытия с улучшенными эксплуатационными свойствами.

Ключевые слова: HVOF, микротвердость, адгезия, структура, фазовый состав, покрытия ZrCN, рентгеновская дифракция.

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