

# Investigation of the Physico-Mechanical Properties of ZrCN Coatings Deposited on the Surface of 65G Steel by the HVOF Method

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## Abstract

Received: August 11, 2026  
Revised: September 24, 2026  
Accepted: September 24, 2026  
Published: September 30, 2026  
<https://doi.org/10.47526/20261/2505-3123.02>

This study investigates ZrCN-based coatings deposited on 65G steel by the high-velocity oxygen-fuel (HVOF) spraying method, with particular attention to the influence of spraying air pressure on coating formation. The study aimed to characterize the resulting microstructure and surface morphology and to assess changes in porosity and microhardness under different spraying conditions. Coatings were deposited using a Termika-3 HVOF system at air pressures of 2.6 and 2.8 bar. Scanning electron microscopy was employed to examine the coating structure and coating-substrate interface, while ImageJ 1.52t was used for quantitative porosity analysis. The coatings exhibited a heterogeneous morphology formed by melted and partially melted particles, with localized pores and surface depressions. The coating-substrate interface remained continuous, without pronounced cracks or delamination. The microstructural observations demonstrated that changes in spraying pressure affected the resulting coating characteristics. Quantitative analysis showed porosity values of 16.8% and 17.48% for the coatings produced at 2.6 and 2.8 bar, respectively. Microhardness measurements yielded values of  $1720 \pm 10$  HV and  $1820 \pm 5$  HV, respectively. These findings provide an experimental basis for selecting HVOF spraying conditions for ZrCN coatings intended to enhance the surface properties of 65G steel.

**Keywords:** ZrCN coatings; 65G steel; HVOF; microhardness; porosity; surface morphology.

## 1. Introduction

Currently, in industry, particularly in the mechanical engineering and automotive sectors, increasing the resistance of steel materials to wear, corrosion, and high-temperature effects is one of the most relevant challenges. Ceramic coatings such as ZrCN (zirconium carbonitride) are widely used to improve mechanical properties, increase wear resistance, and provide protection against chemical effects. However, the formation of ZrCN coatings with high adhesive strength on the surface of 65G steel remains one of the scientific and technological problems that has not yet been fully resolved. The microstructure and degree of porosity of the coating directly affect its microhardness, wear resistance, and adhesion strength. In addition, residual stresses

formed in the coating and the difference in the coefficients of thermal expansion between the coating and substrate materials may cause coating delamination or the formation of cracks [1].

ZrCN coatings are protective ceramic coatings characterized by high hardness, thermal stability, and resistance to wear and corrosion. Zr(C,N) is a solid solution based on ZrC and ZrN, formed as a result of carbon and nitrogen atoms occupying interstitial sites in the crystal lattice. According to the literature [2–4], thermal spraying technologies such as atmospheric plasma spraying (APS), vacuum plasma spraying (VPS), detonation spraying, arc spraying, flame spraying, as well as HVOF are commonly used to produce ceramic-metal-based composite coatings (cermets). HVOF technology is widely used for producing carbide-based protective coatings, and such coatings are characterized by high resistance to abrasive, erosive, and cavitation wear [5–7].

The HVOF method is characterized by a high flame velocity and high flame temperature, with typical values of approximately (~1500 m/s) and (~3000 °C), respectively, depending on the fuel type, oxygen-to-fuel ratio and spray system [8,9]. These conditions ensure that powder particles impact the substrate surface with high kinetic energy and undergo rapid cooling, resulting in the formation of dense, low-porosity coatings with reduced oxidation [10]. In addition, the resulting coatings contribute to increasing the durability of industrial equipment and providing enhanced wear resistance [11,12].

The aim of this study is to comprehensively evaluate the structural characteristics and physico-mechanical properties of ZrCN coatings deposited on the surface of 65G steel using HVOF technology. The study investigates the effect of spraying parameters on the microstructure, porosity level, microhardness, adhesion strength, and tribological properties of the coatings. The obtained results make it possible to identify the relationship between the microstructure and physico-mechanical properties of the coatings, as well as to scientifically substantiate effective HVOF spraying parameters for the formation of ZrCN coatings with high performance characteristics.

## 2. Materials and Methods

ZrCN-based coatings were deposited on 65G steel using a Termika-3 high-velocity oxygen-fuel (HVOF) spraying system (Saint Petersburg, Russia), equipped with a control system for precise adjustment of gas supply pressures. A ZrCN-based powder material with a particle size of 25–40 µm was used to form the coatings. During spraying, the powder was heated in a high-temperature flame flow and directed at high velocity onto the surface of 65G steel, resulting in the formation of a coating layer on its surface.

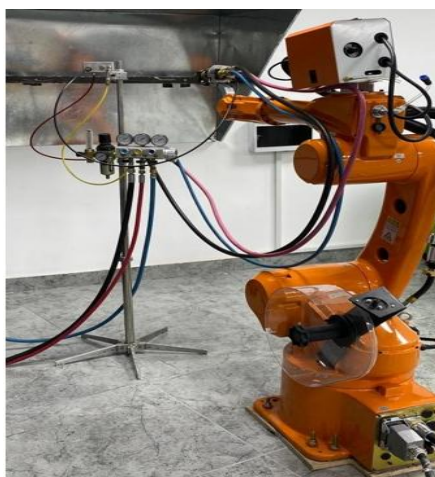


**Fig. 1.** Schematic representation of the operating principle of the HVOF technology [13].

The research object was 65G spring carbon steel. Table 1 presents the chemical composition of 65G steel. Prior to spraying, the substrate surface was mechanically treated (drawn) to remove oxide layers, followed by abrasive sandblasting to improve the adhesion of the coating.

**Table 1.** Chemical composition of 65G steel [14]

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

The obtained coatings were subsequently used as samples for studying their microstructural, porosity, mechanical, tribological properties. ZrCN-based coatings were deposited on the surfaces of 65G steel samples according to the technological parameters presented in Table 2, where the air pressure was intentionally varied from 2.6 to 2.8 bar, while all other spraying parameters were kept constant.

**Table 2.** HVOF spraying parameters used for the deposition of ZrCN coating on the surface of 65G steel

| №        | distance, mm | Fuel  | Air pressure | Oxygen gas | Powder |
|----------|--------------|-------|--------------|------------|--------|
| Sample a | 350          | 3 bar | 2,6 bar      | 3,2 bar    | ZrCN   |
| Sample b | 350          | 3 bar | 2,8 bar      | 3,2 bar    | ZrCN   |

The microstructure, surface morphology, and coating–substrate interface of ZrCN-based coatings deposited on 65G steel by HVOF were examined using a Tescan Vega 4 scanning electron microscope (Brno, Czech Republic) [15,16]. SEM imaging was performed at an accelerating voltage of 25 keV, a working distance of 14.86–14.87 mm, and a magnification of 1000× using BSE and SE detectors. The obtained images were used to evaluate the coating morphology, microstructure, interface condition, and porosity.

The microhardness of the coatings was measured using an HLV-1DT Vickers microhardness tester (Shanghai Hualong Test Instruments Corporation, Shanghai, China) in accordance with the ASTM E384 standard. The measurements were performed using an HLV-1DT microhardness tester. A diamond pyramid indenter with a face angle of 136° was used. During the study, a load of HV<sub>0.5</sub> was applied to the sample surface, with a dwell time of 10 seconds. For each sample, the microhardness measurement was repeated five times, and the arithmetic mean of the obtained values was taken as the final result. The microhardness values were calculated by measuring the diagonal lengths ( $d_1$  and  $d_2$ ) of the indentation impressions [17].

The tribological properties of the coatings were evaluated using a TRB3 tribometer (Anton Paar, Buchs, Switzerland) in accordance with ASTM G99 using a ball-on-disk configuration. During the tests, the coefficient of friction of the coating were determined. A 100Cr6 steel ball with a diameter of 6 mm was used as the counter-body. The tests were conducted under ambient conditions in dry sliding mode. During the study, a normal load of 5 N, a sliding speed of 2 cm/s, and a total sliding distance of 30 m were set. All tests were performed at a temperature of  $25.5 \pm 1$  °C. During the tests, the coefficient of friction and wear parameters were recorded and subsequently used for comparative analysis.

### 3. Results and Discussion

The microhardness of the coatings is one of the key indicators characterizing their mechanical properties. Figure 2 presents the Vickers microhardness values of the initial 65G steel and the ZrCN coatings obtained using the HVOF method. The measurement results showed that the average microhardness of the initial 65G steel was approximately 300 HV. The microhardness of the ZrCN coatings deposited using the HVOF method was significantly higher compared with the initial material, reaching approximately  $1720 \pm 10$  HV for Sample a obtained at an air pressure of 2.6 bar and approximately  $1820 \pm 5$  HV for Sample b obtained at an air pressure of 2.8 bar.

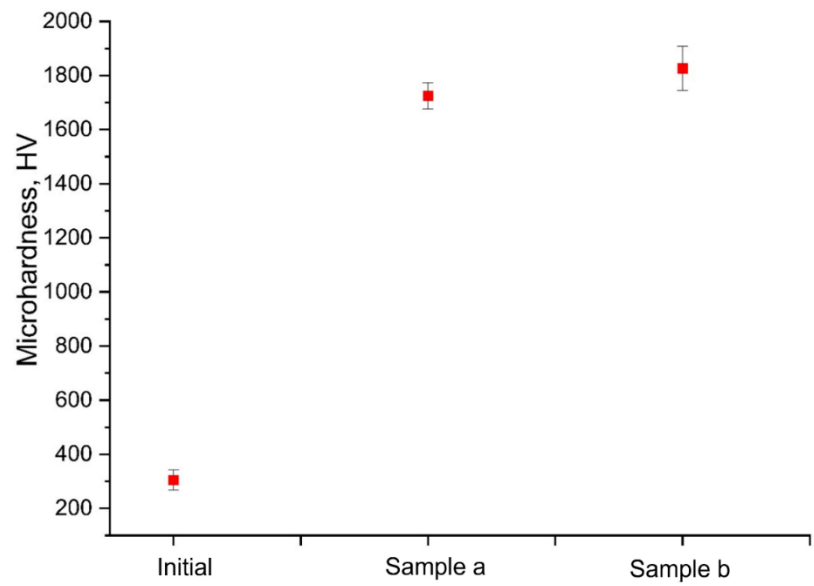


**Fig. 2.** Vickers microhardness of ZrCN coatings obtained by the HVOF method: a — 2.6 bar; b — 2.8 bar.

The obtained results showed that the formation of the ZrCN coating increased the microhardness of the surface layer by approximately 5.7–6 times compared with the initial material. In addition, an increase in air pressure from 2.6 bar to 2.8 bar resulted in an increase in microhardness of approximately 100 HV. This difference can be attributed to the increase in the kinetic energy of the powder particles as a result of the higher air pressure during spraying, their more intensive plastic deformation upon impact with the substrate surface, and the formation of a denser and more homogeneous coating structure.

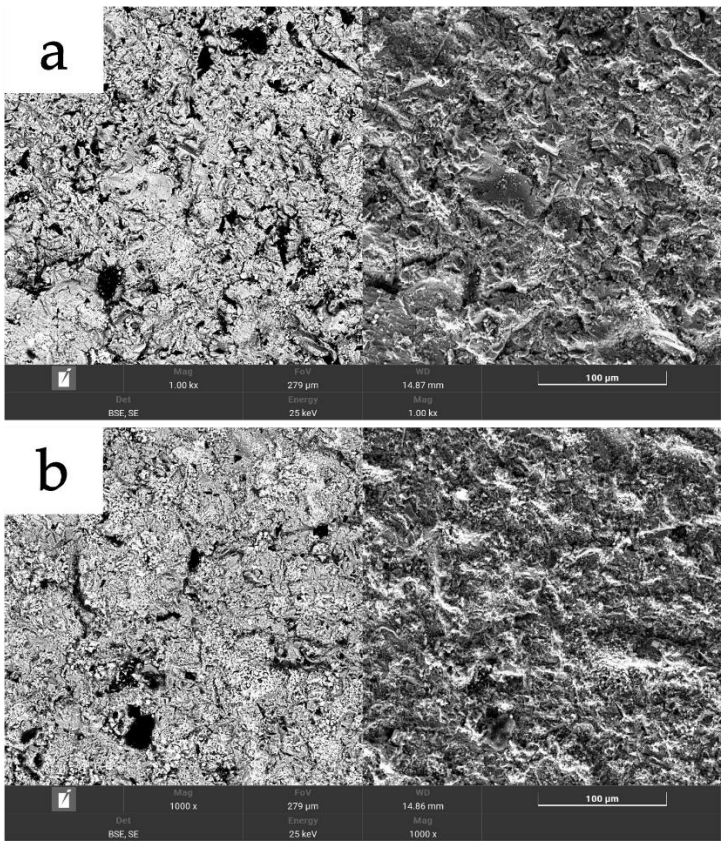


**Fig.3.** SEM images of the surface morphology of ZrCN coatings obtained by the HVOF method.

The SEM images show a clearly pronounced rough morphology of the coating surface. Areas of overlapping and compacted melted and partially melted particles, as well as local micropores and surface depressions, are clearly visible. The distribution of dark-contrast regions indicates the presence of individual pore-like features within the coating structure. At the same time, no widespread system of large and interconnected pores was observed throughout the coating, indicating that its overall structure is relatively dense. No large cracks or signs of coating delamination were detected in the studied samples.

The porosity of the coatings prior to corrosion testing was evaluated from surface micrographs obtained by scanning electron microscopy (SEM) using ImageJ software, version 1.52t. For each coating, five representative SEM images obtained from different areas of the coating surface at the same magnification were analyzed to account for local variations in the coating morphology. The analyzed areas were selected from representative regions of the coating surface, avoiding damaged or non-representative areas. In ImageJ, the porous regions visible in each SEM micrograph were identified by image segmentation, and the ratio of the pore area to the total analyzed image area was calculated. The reported porosity values represent the two-dimensional (2D) surface-area fraction of pores determined from the SEM micrographs and do not represent bulk volumetric porosity. Based on the analyzed areas, the average porosity was 16.80% for Sample a and 17.48% for Sample b, respectively (Figure 4).

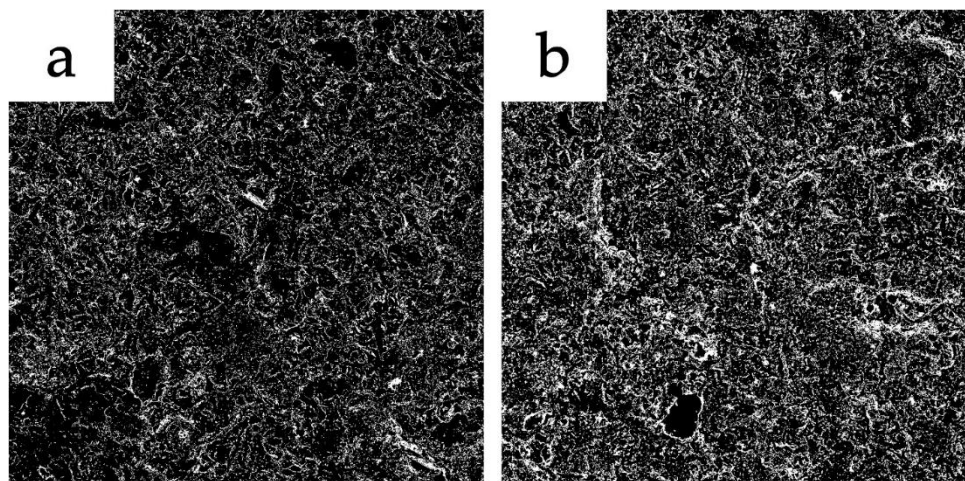


**Fig.4.** Results of the quantitative evaluation of the porosity of ZrCN coatings obtained by the HVOF method using ImageJ software.

According to the SEM image analysis, the two-dimensional pore area fraction was 16.80% for Sample a and 17.48% for Sample b. Thus, an increase in air pressure from 2.6 bar to 2.8 bar resulted in an increase of 0.68 percentage points in the measured 2D pore area fraction. This increase should be interpreted specifically as a change in the pore area fraction observed on the analyzed coating surfaces and does not, by itself, indicate a deterioration of the overall mechanical properties of the coatings. In contrast, the microhardness increased from  $1720 \pm 10$  HV for Sample a to  $1820 \pm 5$  HV for Sample b. Therefore, the observed increase in microhardness cannot be directly attributed to the measured 2D porosity values alone. It may be associated with several factors related to coating formation, including the increased kinetic energy of the powder particles at higher air pressure, their impact and deformation behavior upon contact with the substrate surface, and the resulting local microstructural characteristics of the coating. Further correlation between surface pore area fraction and mechanical properties would require additional three-dimensional porosity characterization and microstructural analysis.

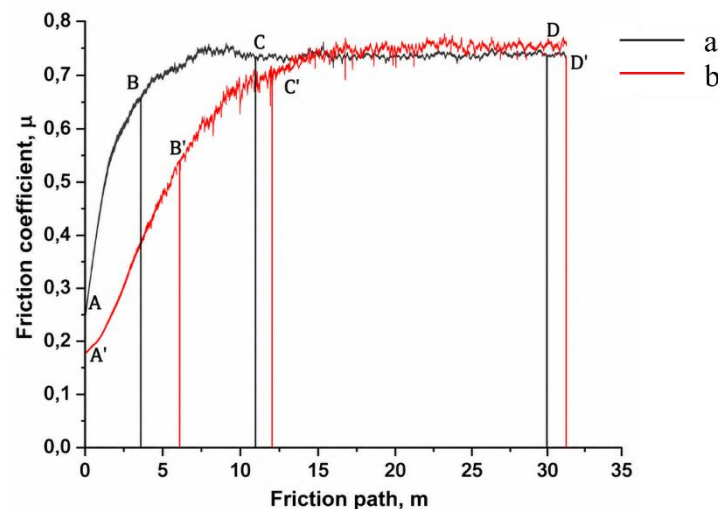


**Fig.5.** The dependence of the coefficient of friction on the sliding distance for ZrCN coatings deposited using the HVOF method is shown. For both samples, the friction process consists of three main stages: the initial running-in stage, the stabilization stage, and the steady-state friction stage.

For Sample a (2.6 bar), during the initial AB stage (0–3.5 m), the coefficient of friction increased rapidly from approximately 0.25 to 0.67. This behavior can be attributed to the initial adaptation between the counter-body and the coating surface, an increase in the actual contact area, and gradual smoothing of surface micro-roughness. During the subsequent BC stage (3.5–11.5 m), the coefficient of friction stabilized within the range of 0.72–0.75, indicating that the coating had reached a relatively stable friction regime. During the CD stage (11.5–30 m), no significant changes in the coefficient of friction were observed, indicating stable friction behavior of the coating under the applied test conditions.

For Sample b (2.8 bar), during the initial A'B' stage (0–6 m), the coefficient of friction gradually increased from 0.20 to 0.55. Subsequently, during the B'C' stage (6–12 m), the coefficient increased to 0.70–0.73 and reached a stable value. During the C'D' stage (12–30 m), the coefficient of friction remained within the range of 0.74–0.76, demonstrating that the coating maintained stable tribological properties over a prolonged period.

A comparison of the results obtained for the two samples showed that the variation in the coefficient of friction of the coating deposited at an air pressure of 2.8 bar was more uniform. This result can be explained by the increase in the kinetic energy of the powder particles due to the higher air pressure during spraying, which promotes more effective plastic deformation of the particles on the substrate surface and the formation of a denser and more uniform structure.

Overall, the tribological test results demonstrated that ZrCN coatings deposited using the HVOF method exhibit high stability under friction conditions and significantly improve the wear resistance of the 65G steel surface.

## 4. Conclusions

The results of the conducted study showed that the formation of ZrCN coatings on the surface of 65G steel using the HVOF method has a significant effect on the structural and mechanical characteristics of the coatings. SEM analysis revealed a complex rough morphology of the coating surface, the interaction of melted and partially melted particles, as well as the presence of local micropores and surface depressions. At the same time, the absence of large cracks and signs of delamination in the studied samples indicates the stable formation of the coating–substrate interface.

Quantitative analysis performed using ImageJ 1.52t software showed that the porosity of Sample a was 16.8%, while that of Sample b was 17.48%. An increase in porosity by 0.68 percentage points was observed with an increase in air pressure from 2.6 bar to 2.8 bar. This result indicates that an increase in spraying pressure does not necessarily lead to a reduction in porosity in all cases. Therefore, it is insufficient to characterize the mechanical properties of the coating solely by its porosity, and other structural factors affecting its formation should also be taken into account.

Microhardness tests showed that the ZrCN coatings exhibited significantly higher hardness compared with the initial 65G steel. At an air pressure of 2.6 bar, the microhardness was  $1720 \pm 10$  HV, whereas at 2.8 bar it reached  $1820 \pm 5$  HV. Thus, an increase in air pressure resulted in an increase in microhardness of approximately 100 HV. The obtained results indicate that the increase in microhardness may be influenced by the increased kinetic energy of the powder particles during spraying, their deformation upon impact with the substrate surface, and the conditions of coating formation.

Despite the slight increase in porosity, the increase in microhardness at the 2.8 bar condition demonstrated that the properties of the coating are not determined by a single parameter alone. This indicates the presence of an interrelationship between spraying pressure, particle velocity, particle deformation, and the formation of the internal structure of the coating during the HVOF process.

Overall, the results showed that the ZrCN coating produced at an air pressure of 2.8 bar exhibited a higher microhardness value than the coating produced at 2.6 bar. Within the investigated experimental conditions, the 2.8 bar condition resulted in a higher surface hardness of the 65G steel. However, since only two air-pressure conditions were investigated, these results should be considered within the studied experimental range. Further evaluation of the coating performance should include additional parameters, such as adhesion strength, coefficient of friction, and quantitative wear characteristics.

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