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Investigation of the Effect of Oxygen Pressure on the Structural–Phase State and Functional Properties of Zr_2CN Coatings Obtained by HVOF Spraying

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Abstract

This study focuses on the analysis of the effect of oxygen pressure on the structural–phase state, microstructure, and microhardness of Zr_2CN coatings produced by high-velocity oxygen fuel (HVOF) spraying. It was established that variation of oxygen pressure within the range of $(4.0–4.5)x10^5$ Pa leads to significant changes in the phase composition, degree of crystallinity, and microstructural features of the resulting coatings. X-ray diffraction analysis revealed the formation of a multiphase system consisting of Zr_2CN, ZrC, ZrN, and ZrO_2 phases, with the carbonitride phase possessing a face-centered cubic structure being dominant. It was demonstrated that the most favorable conditions for coating formation are achieved at an oxygen pressure of $4.3x10^5$ Pa, which ensures stabilization of the Zr_2CN phase and minimizes the content of secondary phases and oxide inclusions. Microstructural investigations (SEM/EDS) confirmed the formation of a dense and homogeneous coating with a uniform distribution of elements and a well-defined interface with the substrate. Mechanical testing showed that the optimal spraying regime provides the best performance characteristics, including enhanced adhesion strength reaching 18.4 MPa. The obtained results demonstrate the key role of oxygen pressure in controlling the structure–property relationship and can be used to optimize HVOF technology for the production of highly effective wear-resistant Zr_2CN-based coatings.

Keywords: U8G carbon stee l; HVOF 2; SEM 3; XRD 4.

1. Introduction

Carbon tool steels, particularly U8G steel, are extensively employed in components subjected to contact loading, as well as abrasive and adhesive wear under cyclic friction conditions. However, their operational lifetime is considerably constrained by severe surface degradation, elevated friction coefficients, localized thermal accumulation within the contact zone, and the rapid initiation and propagation of surface defects [1]. In this context, one of the most effective strategies for enhancing the durability of such components is the application of functional protective coatings that combine high hardness, superior wear resistance, and stable frictional behavior, while preserving the load-bearing capability of the steel substrate [2].

Significant progress in understanding the structure and properties of ZrCN coatings has been achieved through studies on thin-film systems. For example, Cotruț, C.-M. et al. (2013) demonstrated that increasing the nitrogen (N_2) flow during reactive magnetron sputtering promotes the formation of a face-centered cubic (FCC) ZrCN structure. At higher nitrogen concentrations, the emergence of an amorphous phase was observed, which contributes to a reduction in the friction coefficient. Furthermore, alloying with hafnium (Hf) was shown to enhance hardness (by approximately 5 GPa), adhesion strength, corrosion resistance, and biocompatibility of the coatings [3].

The fundamental mechanisms governing carbonitride phase formation have been explored in the work of Zhang, G.Y. et al. (2020), where the synthesis of Zr_2CN via carbothermal reduction was investigated. It was established that the process proceeds through a sequence of intermediate compound formations, ultimately leading to a thermodynamically stable carbonitride phase. Diffusion processes and phase stability were identified as key factors influencing the formation mechanism [4].

Further insights into the structural and mechanical behavior of carbonitride systems have been obtained from studies of bulk materials. In particular, Kuzmenko, E. et al. reported that ZrCN–ZrO_2 ceramics fabricated by spark plasma sintering (SPS) are formed at temperatures around 1562 °C. It was shown that increasing the oxide phase content reduces porosity and enhances material density, thereby improving mechanical performance. The reported properties include a hardness of 17.63 GPa, fracture toughness of 4.22 MPa·$m^{1/2}$, Young's modulus of 426.49 GPa, and strength of 551.41 MPa, while the maximum strength (623.12 MPa) was observed for the ZrN–ZrO_2 system [5]. Additionally, an increase in nitrogen and oxide phase fractions was found to improve oxidation resistance due to the formation of a protective ZrO_2 layer at temperatures exceeding 900 °C.

In this regard, high-velocity oxygen fuel (HVOF) spraying represents a highly effective technique for producing dense, high-performance wear-resistant coatings that satisfy contemporary industrial requirements and sustainability considerations [6, 7].

Accordingly, the present study provides a comprehensive investigation of Zr_2CN coating formation via HVOF spraying, with particular emphasis on the influence of oxygen pressure on microstructure, phase composition, and functional performance. The main objective is to establish quantitative relationships between processing parameters and coating properties, including microhardness, adhesion strength to U8G steel, friction coefficient, and dominant wear mechanisms.

2. Materials and Methods

U8G steel (15·45·3 mm3) was used as the substrate. Before coating, the substrate was sandblasted with white alumina grit, which improved coating adhesion (Table 1) [8].

Table 1. Chemical composition of U8G steel.

C	Si	Mn	Ni	S	P	Cr	Cu
0.8-0.9	0.17-0.33	0.33-0.58	max 0.25	max 0.028	max 0.03	max 0.2	max 0.25

Zr_2CN coatings were produced using the HVOF method using a Termika-3 system. This system is equipped with a control system that regulates the pressure of the supplied gases (propane, oxygen, and air), enabling control of the spraying process parameters.

The schematic and general view of the HVOF system, described in detail in previous studies [9], were used in this work. During operation, the gas components (propane and oxygen) are supplied to the combustion chamber, where they are intensively mixed under elevated pressure, forming a high-temperature gas flow. Zr_2CN powder is injected into the burner at a predetermined velocity and transported by compressed air supplied by a compressor, after which the particles are accelerated and deposited onto the substrate surface.

Spraying was carried out at a fixed distance of 0.3 m and constant propane ($2\ 10^5$ xPa) and air ($3.2x10^5$ Pa) pressures, while oxygen pressure varied from 4.0 to 4.5 $x10^5$ Pa. Three samples (a, b, c) were obtained with different oxygen supply conditions, allowing us to study the effect of this parameter on the structure and properties of the coatings.

Microstructural studies and analysis of the sprayed coatings were performed using scanning electron microscopy (SEM) on a Tescan Vega 4 system, which provides both a surface microstructure overview and cross-section analysis (Brno, Czech Republic, 2024).

The phase composition of the resulting Zr2CN-based coatings was studied using an X'Pert PRO X-ray diffractometer with Cu-Kα radiation (λ = 1.5406 Å), a voltage of 40 kV, and a current of 30 mA. The scanning angle range was from 30.01° to 59.99° with a step of 0.02° and a data accumulation time of 2 s. Diffraction patterns were processed using High Score Plus software with a PDF5+ (2026) database (Malvern Panalytical Empyrean, Almelo, Netherlands, 2009).

To evaluate the adhesion properties of the coatings, tests were performed in accordance with ASTM D4541-22 using a hydraulic adhesion tester Elcometer 510 (Elcometer Instruments, Manchester, UK). The adhesive strength of the coatings was measured under the following conditions: dwell time 0.50 s, target loading rate 1.00 MPa/s, and dolly diameter of 20 mm.

3. Results and Discussion

X-ray diffraction patterns of Zr_2CN coatings (samples a–c) produced by the HVOF method under different oxygen pressures are presented in Figure 1. The analysis indicates the formation of a multiphase system consisting of Zr_2CN, ZrC, ZrN, ZrO_2, and α-Fe phases. The dominant diffraction peaks correspond to a carbonitride solid solution Zr_2CN_γ with a face-centered cubic structure (space group Fm-3m, lattice parameter a ≈ 4.636 Å), confirming the formation of the Zr_2CN phase [7, 10]. The intermediate position of these peaks between the reflections of ZrC and ZrN suggests the substitutional incorporation of carbon and nitrogen atoms within the crystal lattice, consistent with Vegard's law.

In addition to the primary phase, secondary phases of ZrC and ZrN were detected, indicating partial decomposition of the initial powder under high-temperature HVOF conditions (~3000 °C), as well as the non-equilibrium nature of phase formation during rapid cooling. The presence of the ZrO_2 oxide phase, observed as weak diffraction peaks, is attributed to oxidation of particles during their transport within the flame. A pronounced α-Fe reflection reflects the contribution of the substrate, which is particularly significant in sample (a) due to its lower coating thickness and density.

As the oxygen pressure increases from 4.0 to 4.5×10^5 Pa, a systematic evolution of the phase composition and structural state of the coatings is observed. Sample (a) exhibits relatively low intensity of Zr_2CN peaks along with a strong α-Fe contribution, indicating insufficient coating formation. In contrast, sample (b) (4.3×10^5 Pa) demonstrates the highest intensity of carbonitride phase reflections and minimal substrate influence, which corresponds to the formation of a dense, homogeneous structure with a high degree of crystallinity. Further increase in oxygen pressure (sample (c)) leads to an enhancement of oxide-related reflections and a partial decrease in structural order, which is associated with intensified oxidation processes.

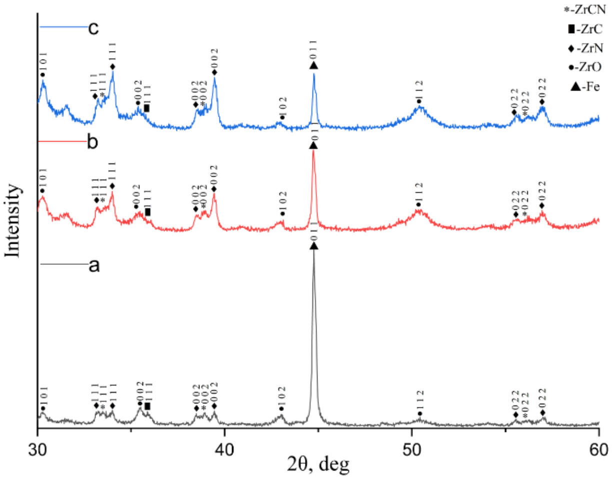

Figure 1. XRD patterns of Zr_2CN coatings obtained at different oxygen pressures: (a) 4×10^5 Pa; (b) 4.3×10^5 Pa; (c) 4.5×10^5 Pa.

A comparative evaluation of elemental distribution within the coatings (samples a–c) demonstrates that the HVOF spraying parameters exert a strong influence on their chemical uniformity and oxidation behavior

(Figure 2). In sample (a), a distinct zirconium diffusion region is observed; however, carbon and nitrogen are distributed non-uniformly, particularly in the vicinity of the coating–substrate interface. The increased oxygen concentration in the upper layer indicates the occurrence of intensive oxidation and the formation of oxide phases.

In contrast, sample (b) exhibits the most homogeneous distribution of the primary elements (Zr, C, and N) throughout the entire coating thickness, along with a minimal oxygen presence. A well-defined iron (Fe) concentration gradient at the interface suggests the formation of a dense and weakly oxidized layer, which contributes to enhanced adhesion and improved protective performance.

Sample (c), on the other hand, is characterized by a higher oxygen content in the upper region of the coating, accompanied by a reduction in the intensity of carbon and nitrogen signals. This behavior indicates partial degradation of the Zr_2CN carbonitride phase and the formation of oxide compounds. Although zirconium remains relatively uniformly distributed, its phase state is likely altered due to oxidation.

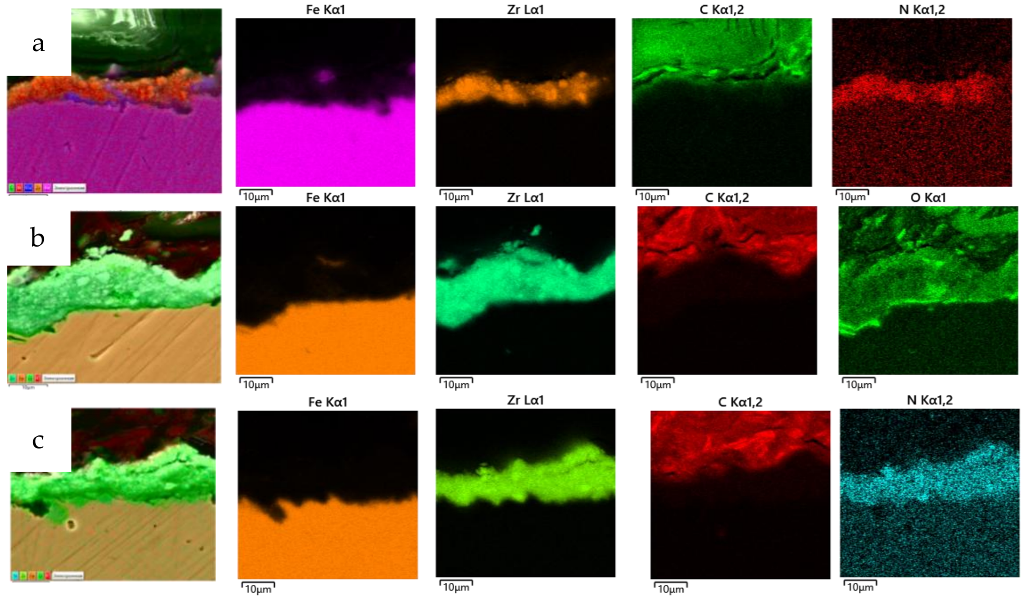

Figure 2. Mapping the distribution of chemical elements on cross-sections of Zr_2CN coatings Sample (a-c).

The adhesion strength results for the Zr_2CN coatings (Figure 3) indicate a pronounced dependence on the HVOF spraying parameters. The measured adhesion values were 14.7 MPa for sample (a), 18.4 MPa for sample (b), and 16.4 MPa for sample (c). The average adhesion strength was 16.5 ± 1.5 MPa, with the standard deviation reflecting variations in coating properties under different spraying conditions.

The maximum adhesion strength was recorded for sample (b), suggesting the formation of the most compact and structurally uniform coating with strong bonding to the substrate. The relatively lower adhesion observed in sample (a) can be attributed to insufficient coating density and the presence of microstructural imperfections. In the case of sample (c), the slight reduction in adhesion strength is associated with an increased fraction of oxide phases.

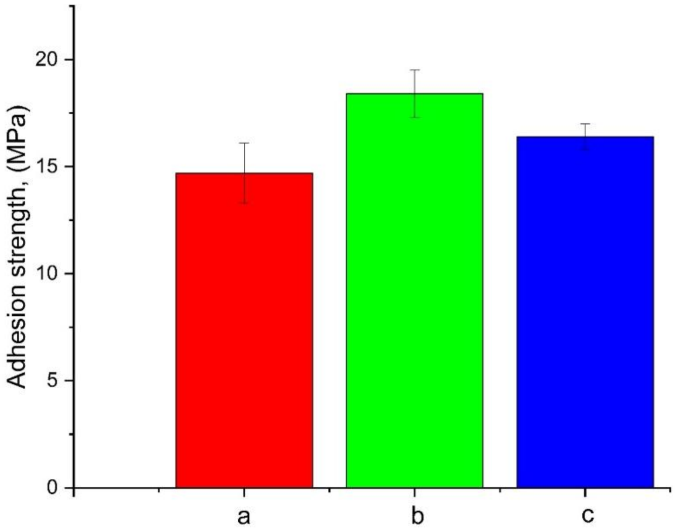

Figure 3. Adhesion strength of Zr_2CN coatings deposited by the HVOF method at different air flow rates.

The maximum adhesion strength was achieved for sample (b), indicating the formation of a dense and structurally homogeneous coating with strong bonding to the substrate. The lower adhesion value observed for sample (a) can be attributed to insufficient coating density and the presence of microstructural defects, whereas the slight reduction in strength for sample (c) is associated with an increased fraction of oxide phases [11, 12].

4. Conclusions

The conducted study demonstrates that oxygen pressure is a critical parameter in the HVOF process, significantly influencing the structural–phase state, microstructure, and performance characteristics of Zr_2CN coatings.

It was established that variation of oxygen pressure within the range of 4.0–4.5x10^5 Pa leads to a systematic evolution of phase composition, degree of crystallinity, and microstructural features of the coatings. X-ray diffraction analysis confirmed the formation of a multiphase system comprising Zr_2CN, ZrC, ZrN, and ZrO_2, with the carbonitride phase exhibiting a face-centered cubic (FCC) structure being predominant. The most favorable conditions for coating formation were achieved at an oxygen pressure of 4.3x10^5 Pa (sample b), where stabilization of the Zr_2CN phase with a minimal content of secondary phases and oxide compounds was observed. Microstructural analysis (SEM/EDS) revealed the formation of a dense and homogeneous coating with a uniform elemental distribution and a well-defined coating–substrate interface.

It was also shown that the optimal spraying regime (sample b) provides the highest adhesion strength, reaching 18.4 MPa. Deviations from this regime result either in insufficient coating formation (sample a) or in intensified oxidation processes accompanied by partial degradation of the carbonitride phase (sample c).

Therefore, an oxygen pressure of 4.3x10^5 Pa can be recommended as the optimal HVOF spraying parameter for producing Zr_2CN coatings with an improved combination of structural and functional properties. The obtained results contribute to a deeper understanding of the process–structure–property relationships and can be applied for the targeted development of wear-resistant coatings intended for operation under high-load conditions.

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