

Comparative analysis of microstructure and selected properties of WC-Co-Cr coatings sprayed by high-velocity oxy fuel on S235 and AZ31 substrates

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The purpose of this work was to carry out comparative studies of WC-Co-Cr coatings deposited using the high velocity oxy fuel (HVOF) method onto two types of substrate material: structural steel S235 and magnesium alloy AZ31. The influence of the substrate material type on the microstructure, phase composition, crystallite size, porosity, Vickers microhardness, instrumental hardness (H_{IT}), Young's modulus (E_{IT}), and fracture toughness was investigated. For both substrates, the deposited coatings deposited were characterized with fine-grained and compact microstructure. The X-ray diffraction (XRD) revealed presence of following phases: WC, W_2C , $Co_{0.9}W_{0.1}$, and $Co_3W_9C_4$. The WC phase was the most desirable and stable one with crystallites were below 100 nm. On the other hand, the size of the W_2C crystallites was below 30 nm. The coatings obtained showed porosity values equal to 2.3 ± 0.4 vol% and 2.8 ± 0.7 vol% for AZ31 and S235, respectively. The average Vickers microhardness for both types of sample was approximately 1200 HV0.3. The average H_{IT} values for carbide particles and metallic matrix were around 29 GPa and 6.5 GPa, respectively. In the case of E_{IT} , it was around 620 GPa and 190 GPa for WC and Co-Cr, respectively. The differences between coatings were negligible. The E_{IT} value for both coatings was equal to 344 ± 11 GPa. The fracture toughness was around $4.5 \text{ MPa} \cdot \text{m}^{1/2}$ in both cases. The investigations revealed that it is possible to replace steel substrate material with a much lighter equivalent, in this case AZ31 alloy, without deterioration of the coating properties.

Keywords: structural steel, magnesium alloy, cermet coatings, HVOF, mechanical properties, microstructure, phase analysis

1. Introduction

Surface engineering is a widespread technology that can be found in many branches of industry [1, 2]. Among the many application methods available, thermal spraying allows for many possibilities in accordance with the requirements for feedstock materials, expected coating properties, process efficiency, and cost [3]. In several applications, the surface of working elements has to meet very difficult and demanding working conditions, for example, high loads, high temperatures, aggressive environments, and so forth. These

conditions require significant resistance on the surface. Materials that can provide good wear, corrosion, and high temperature resistance are mainly cermets [4, 5]. Among these, tungsten carbide with a cobalt and chromium matrix provides very good properties, such as high wear-, corrosion- and cavitation erosion resistance [6–8].

A separate issue is the selection of an appropriate method for incorporating coating into the manufacturing process. One of the most widespread technologies in the field of coatings production is thermal spraying [9, 10]. A comprehensive description of the methods of thermal spraying, its properties, and possible applications can be found, for

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example, in Galedari et al. [11], Joshi [12], and Latka et al. [13].

Because of their very interesting properties, thermalsprayed cermet coatings, mainly produced by the high-velocity oxy-fuel (HVOF) method [14, 15], have been intensively investigated for many years. Nevertheless, these surface deposits have mainly been applied to different steel substrates. Given the current trends of the industry, weight reduction plays a significant role [16, 17]. Therefore, more and more often, other metallic alloys are used as the base material and substrate for cermet coatings [18, 19]. Therefore, the a group of lighter materials, namely magnesium alloys, have to be taken into consideration. This materials group exhibits interesting properties, but a wide expansion of its use is blocked in many industrial branches as a result of important drawbacks. The strengths and weaknesses of magnesium alloys are described in detail by Tan and Ramakrishna [20]. The possibility of widespread use of magnesium alloys in industry requires significant improvement in the properties of the material's surface. Recently, a detailed description of Mg-based surface modification methods and their characterization was given by Morelli et al. [21]. Regarding the area of thermal spraying, the most promising method for cermet coating deposition on magnesium alloy is HVOF. There are only a few examples of investigations are found in the literature [22–24]. Taking into consideration that weight reduction would be the main reason for replacing steel as the base material, it is advisable to carry out investigations on the influence of the type of substrate on the mechanical and functional properties of manufactured coatings. It is particularly important given that, for steel substitution, mass reduction needs to be accomplished without changing operational conditions. In the case of high requirements for wear, corrosion, and oxidation resistance, the tungsten carbide (WC)-based powder with a Co matrix and a Cr addition is often used [25, 26].

In this paper, the influence of the substrate type on the microstructure and mechanical properties of the coating was examined. The HVOF cermet coatings were deposited on S235JR structural steel as well as on AZ31 magnesium alloy. The differences

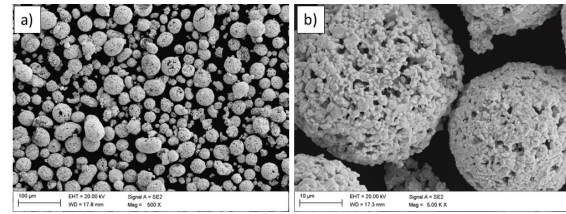


Fig. 1. Morphology of the initial feedstock powder: (a) standard view, (b) detailed view

in thermal conductivity and melting point of the different substrates were also taken into account in the selection of the preliminary process parameters in order to obtain a coating of good quality. The aim of this study was to compare manufactured coating deposits in terms of their phase composition, porosity level, and mechanical properties in order to indicate a potential substrate material that could be a substitute for the conventional steel substrate.

2. Materials and methods

2.1. Substrate and feedstock materials

Two types of metallic materials, structural steel S235JR and magnesium alloy AZ31, with a thickness of 5 mm and a diameter of 25 mm, were used as substrates. Before the deposition process, the substrates' surfaces were sand-blasted with corundum powder (F40 according to the FEPA standard) and cleaned in an ultrasonic bath with ethanol.

The feedstock material was a commercially available powder Amperit 554.071 (Höganäs): WC-10Co-4Cr (wt%) with an apparent density in the range 4.7 to 5.6 g/cm³. According to the manufacturer's declaration, the particle size range was $-45 + 15 \mu\text{m}$, and the delivery condition was agglomerated and sintered. The SEM image confirmed this information (Fig. 1). Additionally, the particle size distribution was assessed by the laser diffraction method using PSA 1190 (Anton Paar). The average particle size of the initial feedstock was equal to 34.2 μm .

2.2. Deposition process

The manufacturing process of the cermet coatings on the different substrate types was realized

Table 1. HVOF process parameters and sample code

Sample	Medium feed rate				Spray distance mm
	Kerosene l/min	Oxygen l/min	Nitrogen l/min	Powder g/min	
SBS ¹	0.380	950	12	70	300
MBS ²	0.435	900			360

¹ SBS – steel-based substrate; ² MBS – magnesium-based substrate

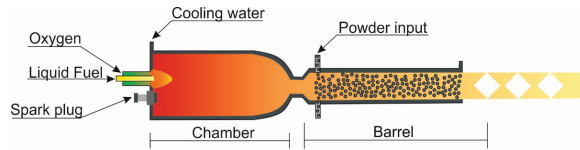


Fig. 2. Scheme of the HVOF deposition method

using a JP 5000 gun from the TAFE system. This gun operates with kerosene as a liquid fuel medium, and the powder is injected in radial mode. The scheme of this system is given in Figure 2.

The process parameters were optimized for steel and magnesium alloy separately (Table 1). As can be seen, the two factors are clearly different. The spray distance for AZ31 is longer because of the flammability of the magnesium, and the oxygen to fuel ratio ($\lambda_{O/F}$) [27] is equal to 1.22 and 1.02 for steel and magnesium alloy substrate, respectively. The ratio is connected with the specific properties of each of the substrate materials.

2.3. Microstructure characterization

The topography of manufactured cermet coatings was observed using scanning electron microscopy (SEM) (Supra 35, Zeiss). The surface roughness in the as-sprayed state was measured with the use of the MarSurf PS10 profilometer (Mahr) according to the ISO 4288 standard. The average value of the R_a , R_p , R_v , and R_z indicators as well as standard deviation values from five measurements were calculated. In order to carry out microstructure investigations, samples were cut on a precision cutter, then ground with SiC papers up to a grit size of 2500 and polished with a 3- μ m diamond suspension. The microstructure of deposited samples from these prepared cross sections as well as the coating's thickness were examined using SEM apparatus.

Ten measurements were taken, and the average value with standard deviation was calculated. The coating's porosity was also assessed on the cross sections, according to the ASTM E2109-01 standard. The calculations, based on 20 SEM images at 1000x magnification for each coating, were carried out using ImageJ software (version 1.50i). The phase composition was estimated by X-ray diffraction (XRD) using a D8 Advance diffractometer (Bruker) with a Cu-K α cathode (wave length equal to 0.154 nm). The operating current and voltage were equal to 40 mA and 40 kV, respectively. The measurements were carried out for a 2θ angle in a range from 20° to 100° with 0.60°/min scan rate and 0.02° scanning step. The phases were fitted and identified using DIFFRAC.EVA software with ICDD PDF#2 database.

2.4. Mechanical properties estimation

The Vickers microhardness test of the coatings was carried out according to the ISO 4516 standard with values of maximum load and dwell time equal to 2.94 N (HV0.3) and 15 s, respectively. For each coating, 15 imprints were made and then the average values with standard deviation were calculated, and in both coatings the fine structure was obtained. For this purpose, the instrumental indentation technique (IIT) was used in order to measure the hardness of the carbide particles and metallic matrix. The instrumental hardness (H_{IT}) assessment was carried out on the samples' cross sections, according to the ISO 14577-4 standard, using NHT³ nanoindenter (Anton Paar) with the Berkovich indenter. The value of the maximum load was equal to 50 mN. Ten imprints for carbides and matrix area were carried out for both samples. The Oliver-Pharr method was used in order to calculate the instrumental elastic modulus (H_{IT}) [28].

Table 2. Results for surface roughness of the as-sprayed coatings

Sample	Ra	Rp	Rv	Rz
SBS	3.36 ± 0.23	10.20 ± 2.14	7.95 ± 1.48	18.10 ± 3.15
MBS	4.28 ± 0.24	11.84 ± 2.47	9.92 ± 1.83	21.77 ± 3.51

Using the same apparatus, it was also possible to assess the instrumental elastic modulus (E_{IT}) of deposited coatings. The value of maximum load ranged from 100 mN to 500 mN, and E_{IT} estimation was made based on methodology proposed by Tricoteaux et al. [29] and modified by Łatka et al. [30]. For each sample, five repetitions were carried out. In the case of EIT assessment for tungsten carbide particles and the metallic matrix, the maximum load ranged from 25 mN up to 100 mN.

The fracture toughness (K_{IC}) of the manufactured coatings was estimated using the indentation method, with the Vickers indenter, according to the ASTM C1421 standard. The value of the maximum load was equal to 98.1 N, and 10 imprints were made for each sample. The average value of K_{IC} was calculated with Equation (1):

$$K_{IC} = 0.079 \cdot \left(\frac{P}{a^{3/2}} \right) \cdot \log \left(\frac{4.5 \cdot a}{c} \right) \quad (1)$$

In Equation (1) the elements are as follows: P is maximum load, N; a is indentation half diagonal, m; c is crack length from the center of the indent, m. This model was proposed by Evans and Wilshaw [31] and adapted to HVOF coatings by Liu et al. [32].

3. Results and discussion

3.1. Coating morphologies and microstructures

The topography of manufactured coatings is presented on the top-surface SEM images shown in Figure 3. In general, it is a typical morphology for HVOF coatings [33, 34]. The detailed observations confirmed a generally smooth surface with some minor irregularities. The presence of the defects could be related to phenomena occurring in the spray process. Carbide particles remain solid in the flame because of their high melting point

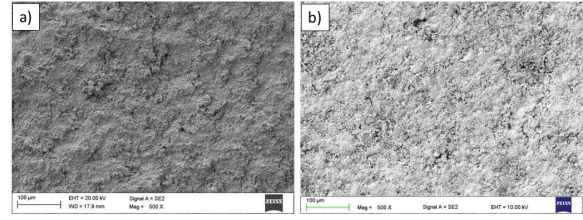


Fig. 3. The surface topography of sprayed coatings: (a) MBS, (b) SBS

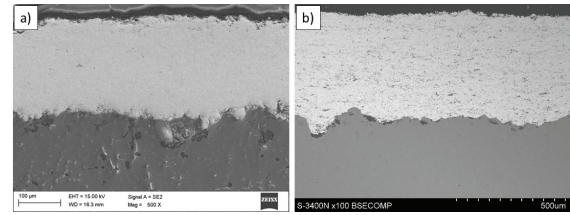


Fig. 4. The SEM images of the coatings' cross-sections at low magnification (500x): (a) MBS and (b) SBS

and relatively high heat capacity, whereas metallic matrix becomes liquid [35]. Moreover, some partially melted or even unmelted fine particles could be seen. This could be related to a lower particle inertia moving them on the flame's periphery [4].

The results of surface roughness measurements expressed by the most important parameters are collected in Table 2. The data obtained are in good agreement with the literature [36, 37]. It should be stressed that in the vast majority of applications the surface is mechanically treated. On the other hand, when the surface roughness for as-sprayed surfaces is lower it facilitates the finish machining of coatings.

The microstructure observation on SEM images was carried out at low (500x) and high (10,000x) magnification. In Figure 4, it can be seen that coatings exhibit dense, compact and uniform structure that is typical for the HVOF process [38, 39]. Moreover, at the interface the tightly adhering

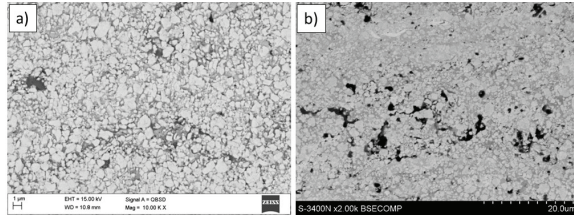


Fig. 5. The SEM images of the coatings' cross-sections at high magnification (10,000x): (a) MBS and (b) SBS

coating can be seen. It is the result of proper substrate preparation and reflects the advantages of the HVOF process, which is the high kinetic energy of the molten particles and/or highly plasticized powder particles [40, 41]. It strengthens mechanical bonding between coating and substrate and improves adhesion.

The coatings' microstructure observed at high magnification (Fig. 5) also confirms a good quality of deposits. Uniformly distributed carbides particles in metal matrix are wettable by the Co-Cr mixture. Nevertheless, there are some minor material discontinuities, such as cracks and pores. These defects could be a result of thermal stresses that occurred during the deposition process and also the fact that the feedstock particles stayed in the flame for a relatively short time [42, 43].

On the coatings' cross sections, additional analyses were performed in order to estimate porosity. The results show that MBS exhibits a slightly lower porosity value (2.3 ± 0.4 vol%) than that for SBS (2.8 ± 0.7 vol%). For both samples, these values are similar to the data found in the literature [44, 45]. Another issue is the dimensions of the observed pores. Mostly, they are less than $1 \mu\text{m}$. Such fine pores in combination with compact structure could be beneficial from the point of view of an improvement in corrosion resistance [46].

The analysis and results above are strongly connected with process parameters (Table 1). In the HVOF method, increasing the oxygen and fuel flow rate results in an increase of the particle velocity and temperature. This trend, however, is only for "critical O_2 flow rate," which is about 850 L/min. Then, with increasing flow rate, the particle temperature decreases while particle velocity continues

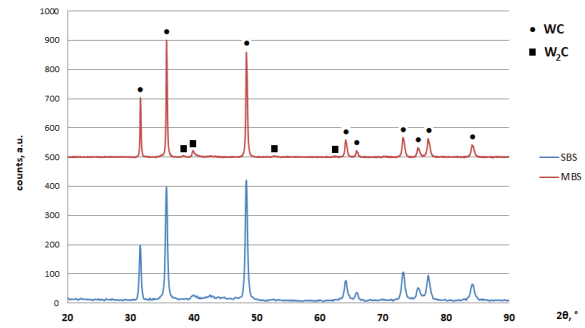


Fig. 6. XRD patterns for HVOF-sprayed coatings

to increase. In case of kerosene, the "critical flow rate" is about 0.385 L/min, but after crossing that point, the changes differ, as in case of oxygen flow rate. Particle temperature decreases, but particle velocity remains constant [47]. Taking into account the kerosene-oxygen mixture, the excess oxygen cools down the flame, thus reducing particle temperature. On the other hand, particles move in the flame at a higher velocity (for greater oxygen flow rate), which results in reducing the residence time in the HVOF flame [48]. This could provide an explanation for why the porosity of the SBS sample is slightly higher than that of MBS.

The XRD diagrams for deposited coatings are presented in Figure 6. In both cases, there are two main phases: WC and W_2C , except for some minor amounts of $\text{Co}_{0.9}\text{W}_{0.1}$ and $\text{Co}_3\text{W}_9\text{C}_4$ that were detected. Similar results were obtained by Pulsford et al. [49] and Komarov et al. [50].

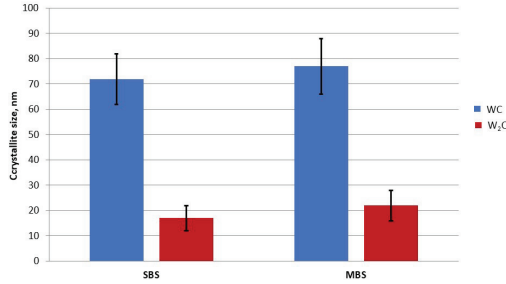
Additional information, which can be extracted during XRD diagram analysis, is the carbide retention index (CRI). More details about this indicator can be found in articles by Liphout and Sato [51] and Górník et al. [52]. This factor is calculated with Equation (2):

$$CRI = \frac{I_{WC}}{I_{WC} + I_{W_2C} + I_W} \quad (2)$$

where the intensity I of the diffraction peaks corresponds to WC, W_2C , and W measured at the positions $2\theta = 35.7^\circ$, $2\theta = 39.8^\circ$, and $2\theta = 40.5^\circ$, respectively [53]. The calculated values of the CRI are collected in Table 3. The results are similar to findings reported by Picas et al. [54].

Table 3. Results for CRI calculations

Sample CRI value	
SBS	0.915
MBS	0.928

Fig. 7. Results of crystallite size calculations for WC and W₂C phases in the sprayed coatings

Determination of the crystallite sizes was carried out on the basis of the width of the diffraction curves of the crystal plane measurements and Scherrer formula [55] according to Equation (3):

$$D_p = \frac{0.94 \cdot \lambda}{\beta \cdot \cos \theta_B} \quad (3)$$

In Equation (3), the elements are as follows: D_p is the crystallite size; λ is the wavelength of X-ray radiation; β is the full width at half maximum (FWHM) of the peak intensity; θ_B is the Bragg angle of reflection of a specific crystal plane.

Figure 7 presents the calculated crystallite size for the main phases (WC and W₂C). The values are slightly lower for the SBS sample. However, the differences are within the value of the standard deviation. In the case of WC, the crystallite size is less than 90 nm, whereas for W₂C it is less than 30 nm. These values are similar to those obtained by Tillmann et al. [56].

3.2. Mechanical properties of sprayed coatings

The results of microhardness measurements of the manufactured coatings confirmed the established hypothesis that proper selection of HVOF parameters results in no change in the mechanical properties, regardless of the substrate material. The results obtained are highly correlated with the

Table 4. The IIT results for carbide particles and metallic matrix in sprayed coatings

Sample	H _{IT} (WC) GPa	E _{IT} (WC) GPa	H _{IT} (Co-Cr) GPa	E _{IT} (Co-Cr) GPa
SBS	29.14 ± 1.91	622 ± 17	6.52 ± 0.47	192 ± 9
MBS	28.65 ± 1.94	624 ± 18	6.46 ± 0.42	193 ± 8

porosity level and are as follows: 1192 ± 114 HV0.3 and 1238 ± 105 HV0.3 for SBS and MBS, respectively. These values are close to the those found in the literature [57, 58].

The IIT results for carbide particles and the metallic matrix are presented in Table 4. The differences are within the range of the standard deviation. Values obtained are similar to those in the literature [59, 60]. Slightly lower E_{IT} values for tungsten carbides could be a result of the spraying process and, in part, the decarburization phenomenon [61]. On the other hand, the E_{IT} values for sprayed coatings was practically identical, and it was equal to 344 ± 11 GPa. Results that were congruous with these were obtained by Culha et al. [62] and Matikainen et al. [63].

In order to ensure high wear resistance, which is a fundamental property of the WC-Co-Cr coatings, not only high hardness but also high fracture toughness (K_{IC}) is required. The results for deposited coatings are as follows: 4.72 ± 0.68 MPa · m^{1/2} and 4.57 ± 0.75 MPa · m^{1/2} for MBS and SBS, respectively. These values are in strong agreement with literature data [64, 65]. The differences in K_{IC} values for the coatings investigated could be a result of different process parameters. It is well known that fracture toughness depends on, among other things, oxidation decarburization [66]. In current studies, the flame for the SBS sample exhibits a more oxidative nature. The flame used for MBS limits the formation of a brittle W₂C phase.

4. Conclusions

In this study, commercially available WC-Co-Cr powder was deposited on two types of metallic

substrates, structural steel and magnesium alloy, using the HVOF method. The main goal of this investigation was to check the possibility of substituting the substrate material without causing deterioration of the coating properties. In order to prove this hypothesis, the deposits were investigated in terms of their microstructure, phase composition, porosity level, and mechanical properties. The main findings were as follows:

- The fundamental issue is proper selection of the process parameters in terms of the substrate and feedstock materials. For steel substrate, the flame was more oxidizing; whereas for magnesium alloy, it should be almost neutral. Moreover, the spray distance was longer for AZ31 because of the flammability of magnesium. This affected the slightly lower porosity level for the MBS sample.
- XRD analysis revealed two main phases in both coatings: WC and W_2C . In addition, minor amounts of $Co_{0.9}W_{0.1}$ and $Co_3W_9C_4$ were detected. The CRI value was insignificantly different, and it was around 0.92 for both. The crystallites were less than 100 nm and below 30 nm for WC and W_2C , respectively.
- Assessment of the mechanical properties revealed that the differences between coatings are generally within the standard deviation. These observations confirm that the substrate material can be changed without causing the coating to deteriorate.

Acknowledgments

The authors gratefully acknowledge Dr. Marcin Godzierz from Centre of Polymer and Carbon Materials Polish Academy of Science for help in the phase analysis assessment.

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Received 2024-02-13

Accepted 2024-03-17