

# Effect of spray distance on the microstructure and corrosion resistance of WC-based coatings sprayed by HVOF

Ewa JONDA<sup>1</sup> , Leszek ŁATKA<sup>2</sup>, Artur MACIEJ<sup>3</sup>, Marcin GODZIERZ<sup>4</sup>,  
Klaudiusz GOŁOMBEK<sup>5</sup>, and Andrzej RADZISZEWSKI<sup>6</sup>

<sup>1</sup> Silesian University of Technology, Faculty of Mechanical Engineering, Department of Engineering Materials and Biomaterials,  
ul. Konarskiego 18a, 44-100 Gliwice, Poland

<sup>2</sup> Wrocław University of Science and Technology, Faculty of Mechanical Engineering, Department of Metal Forming, Welding and Metrology,  
ul. Łukasiewicza 5, 50-371 Wrocław, Poland

<sup>3</sup> Silesian University of Technology, Faculty of Chemistry, Department of Inorganic and Analytical Chemistry and Electrochemistry,  
ul. Krzywoustego 6B, 44-100 Gliwice, Poland

<sup>4</sup> Polish Academy of Sciences, Centre of Polymer and Carbon Materials, ul. M. Curie-Skłodowskiej 34, 41-819 Zabrze, Poland

<sup>5</sup> Silesian University of Technology, Laboratory of the Testy Materials, Silesian University of Technology,  
ul. Konarskiego 18a, 44-100 Gliwice, Poland

<sup>6</sup> "RESURS" Company, A. Radziszewski, ul. Czarodzieja 12, 03-116 Warszawa, Poland

**Abstract.** Cermet coatings provide protection against an aggressive operating environment of machine and device elements, such as corrosion, wear or high-temperature conditions. Currently, WC-based cermet coatings are frequently used in different industry branches. In this work, conventional WC-based powders (WC-Co and WC-Co-Cr) were sprayed with high velocity oxy fuel (HVOF) onto AZ31 magnesium alloy with different spray distances (320 and 400 mm). The research aimed to investigate the effect of the spray distance on the microstructure of the coatings, phase composition and electrochemical corrosion resistance. Results revealed that a higher spray distance results in greater porosity, 1.9% and 2.3% for 320 mm and 2.8% and 3.1% for 400 mm in the case of WC-Co and WC-Co-Cr coatings, respectively. Also, an impact on coatings microhardness was observed, c.a. 1300 HV0.3 for shorter spray distances, whereas for longer ones it was less than 1100 HV0.3. The corrosion resistance estimated in potentiodynamic polarization measurements was the best for WC-Co-Cr coating deposited from the shorter spray distance, corrosion current density was equal to  $2.9 \mu\text{A}\cdot\text{cm}^{-2}$  and polarization resistance was equal to  $8424 \Omega\cdot\text{cm}^2$ .

**Key words:** WC-based powders; AZ31 magnesium alloy; high velocity oxy fuel; microstructure; corrosion resistance.

## 1. INTRODUCTION

Due to the low value of specific gravity and high specific strength, magnesium alloys are increasingly used in a wide range of industry branches, among others, in automotive, aerospace, chemical, shipbuilding and electronics [1–3]. However, their application is limited by low corrosion-, erosion- and abrasive-wear resistance, as well as low hardness [4–6]. The corrosion mechanism of magnesium alloys depends on the type of alloy, impurities from the production process and the possibilities of its potential applications, among others. [7]. In surface engineering, there are two main methods of improving the corrosion resistance of the surface of magnesium alloys by modifying their properties: physical or chemical techniques [8, 9]. One of them is manufacturing protective and/or regenerative coatings. However, in such a case, special atten-

tion should be paid to the problem of the flammability and high plasticity of magnesium alloys [10, 11]. Therefore, it is necessary to select an appropriate technology of surface engineering which allows for the production of a dense, compact and well-adhered coating under relatively low-temperature conditions affecting the substrate [12]. The method in the field of thermal spraying which meets the above requirements is high velocity oxygen fuel (HVOF) spraying [13, 14]. One of the most significant advantages of this method is the possibility to manufacture ceramic, carbide as well as cermet coatings of almost unrestricted chemical and/or phase composition [15–17]. In the area of developing modern techniques of enhancement of the surface properties of machine elements, this method is a good alternative to other methods in the area of thermal spraying and is mainly used to make coatings with high abrasion resistance, as well as high temperature and corrosion resistance [18–20]. Nevertheless, such properties could be achieved only with correctly selected parameters of the thermal spraying process [21, 22]. It is also important that in this scientific area, there is only a small number of papers dealing with research on the production of

\*e-mail: ewa.jonda@polsl.pl

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the above-mentioned coatings deposited on magnesium alloys, and the authors' own research constitutes a certain knowledge base, the aim of which is to fill this gap in the literature [23,24]. Therefore, this paper focuses on the impact of one of the most significant process parameters, the spray distance, on the microstructure and corrosion resistance of the manufactured coatings. The comparison of the two types of cermet materials was also highlighted in the paper.

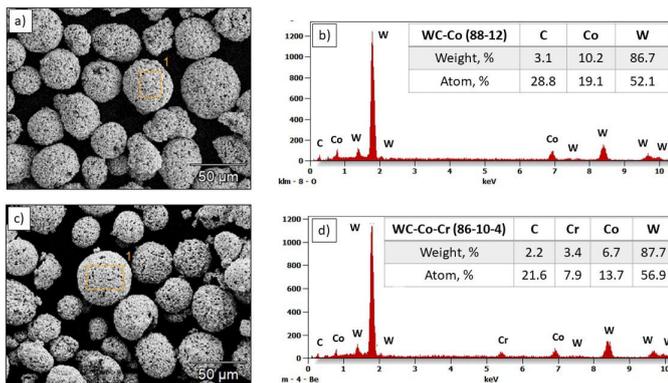
## 2. MATERIALS AND METHODS

### 2.1. Feedstock

In this paper, two commercial powders (from Höganäs) were used, namely:

- Amperit 518.074: WC-12Co (wt.%)
- Amperit 558.074: WC-10Co-4Cr (wt.%)

The delivery state for both powders was agglomerated and sintered. Moreover, the range of particle size declared by the manufacturer was  $-45 + 15 \mu\text{m}$  for both feedstocks. The SEM observations confirmed that particles have a spherical shape (Fig. 1) which improves their flowability during thermal spraying.



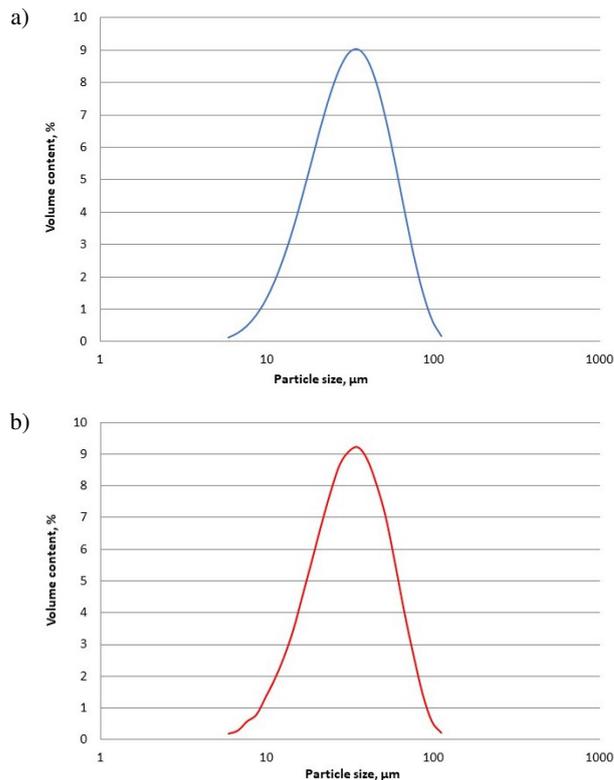
**Fig. 1.** Morphology of WC-Co (a), and WC-Co-Cr (c), the results of chemical analysis (b) from point (a) and (d) from point (c) of the powders, (SEM)

Moreover, the examination of the average particle size of the powders was performed using Fritsch Analysette 22 Micro Tec plus (Germany). The average particle size of the powders is given in Fig. 2. It can be seen that the average particle size  $d_{V50}$  of both used powders is similar ( $34 - 35 \mu\text{m}$ ).

### 2.2. Deposition process

In this study, AZ31 magnesium alloy (with 5 mm thickness) was used as a substrate material. Before the deposition process samples were sandblasted (final  $R_a = 3.2 \mu\text{m}$ ) and cleaned with ethanol. A spray system JP 5000 TAFE (Indianapolis, USA) from RESURS (Warsaw, Poland) was employed for the deposition of the coatings. The spray parameters are summarized in Table 1.

In current studies, the variable factor was the spray distance which is one of the key process parameters. The sample code with the values of the variable process parameters is presented in Table 2.



**Fig. 2.** Particle size distribution of powders used for spraying: WC-Co (a) and WC-Co-Cr (b)

**Table 1**  
Spray parameters

Kerosene (l/h)	26.1
Oxygen (slpm)	900
Nitrogen (slpm)	12
Powder feed rate (g/min)	70
Water flow (slpm)	23

**Table 2**

The sample code and variable parameter values

The feedstock powder	Spray distance, mm	
	320	400
	Sample code	
WC-Co	C1-D1	C1-D3
WC-Co-Cr	C2-D1	C2-D3

### 2.3. Characterization of coatings

After HVOF thermal spraying, the standard metallographic procedure was applied. The cross sections of the coated samples were prepared by cutting, embedding in cold mounting resin and then grinding and polishing. Scanning electron microscope with secondary electron and backscattered detectors (Supra 35,

Zeiss, Oberkochen, Germany) was used for microscopic investigations and fracture morphology. The chemical composition was analyzed by EDS (energy dispersive X-ray spectroscopy). The porosity of the coatings was measured by means of an image analysis software ImageJ open coating according to ASTM E2109 – 01 standard. The cross sections of obtained samples were observed by a digital light microscope Keyence VHX6000 (Keyence International, Mechelen, Belgium). Twenty measurements at the magnification of 1000x were carried out at random locations. Then, the average thickness, as well as standard deviation values were calculated.

The X-ray diffraction (XRD) studies were performed using the D8 Advance diffractometer (Bruker, Karlsruhe, Germany) with Cu-K $\alpha$  cathode ( $\lambda = 0.154$  nm). The copper tube was operating at 40 kV voltage and 40 mA current. The samples were examined in the range of  $20^\circ$  to  $120^\circ 2\theta$ , with a scanning step of  $0.02^\circ$  and a scan rate of  $0.60^\circ/\text{min}$ . Fitted phases were identified using DIFFRAC.EVA program equipped with ICDD PDF#2 database. The exact lattice parameters of the fitted phase were calculated using Rietveld refinement in TOPAS 6 program, using a pseudo-Voigt function in the description of diffraction line profiles. As mathematical factors of calculated for experimental pattern fit, the  $R_{wp}$  (weighted-pattern factor) and S (goodness-of-fit) parameters were used.

The surface roughness of the deposited coatings was measured according to the ISO 4288 standard using a profilometer MarSurf PS10 (Mahr, Gottingen, Germany). For each sample, five measurements of  $R_a$  and  $R_z$  parameters were conducted. Then the average and standard deviation values were calculated.

Vickers microhardness was estimated with the value of maximum load equal to 2.94 N (HV0.3) according to the ISO 4516 standard and was developed to evaluate the hardness of the sprayed coatings. At least ten imprints were conducted on the coating cross-section. The HV-1000 hardness tester (Sinowon Innovation Metrology) was used. The average value of microhardness, as well as the standard deviation, was calculated.

The corrosion resistance investigations of the sprayed AZ31 alloy were realized by the potentiodynamic Tafel method using an AUTOLAB PGSTAT100 potentiostat – galvanostat (Metrohm Autolab BV, The Netherlands). The corrosion resistance assessment was conducted in a 3.5% solution of NaCl at a temperature equal to  $25^\circ\text{C}$ . Before the determination of the polarization and open circuit potentials (EOC), all samples were stabilized at a time equal to 3600 s. The measurements of the linear sweep voltammetry (LSV) were estimated at potential values in the range of EOC from  $-150$  mV up to  $+150$  mV. The scan rate was equal to  $0.167$  mV $\cdot$ s $^{-1}$ . The achieved voltammograms were analyzed by NOVA 2.1 software in order to determine corrosion properties, such as polarization resistance ( $R_p$ ), corrosion current density ( $j_{\text{corr}}$ ), and corrosion potential ( $E_{\text{corr}}$ ). The electrochemical cell used in the measurements was built in the double-walled (thermostatic) mode with a three-electrode configuration. A saturated calomel electrode (SCE) was used as the reference. Additionally, a platinum mesh was used as the counter electrode. For comparison, analogical measurements were also conducted for not coated AZ31 magnesium alloy.

### 3. RESULTS AND DISCUSSION

#### 3.1. Microstructure of the coatings

The observations of the coating topography revealed a typical morphology of HVOF-sprayed coatings [25,26]. In general, the surface is relatively smooth, with some irregularity which could be observed (Fig. 3) probably due to the fact that the hard carbide particles did not melt in the flame [27]. Some differences in the surface roughness of manufactured coatings could be observed. It is an effect of the spray distance. Similar dependence – an increasing roughness with elongation of the stand-off distance was observed in [28].

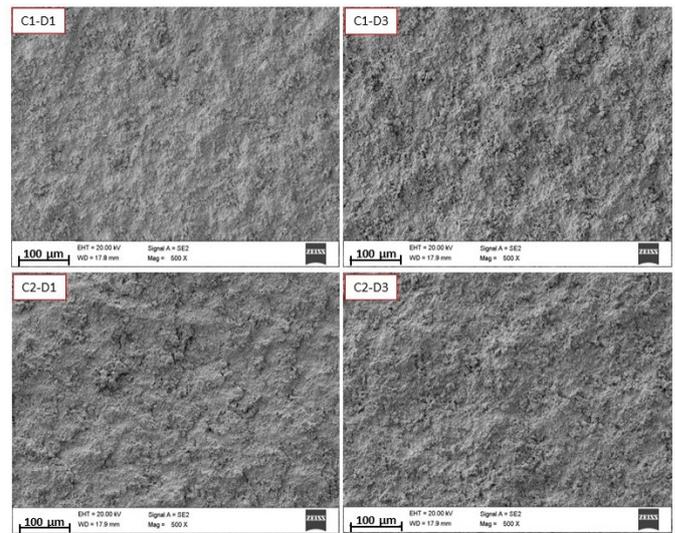
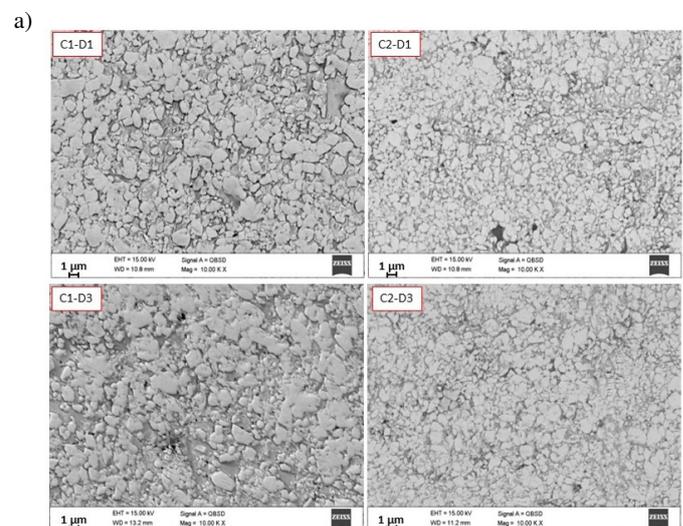
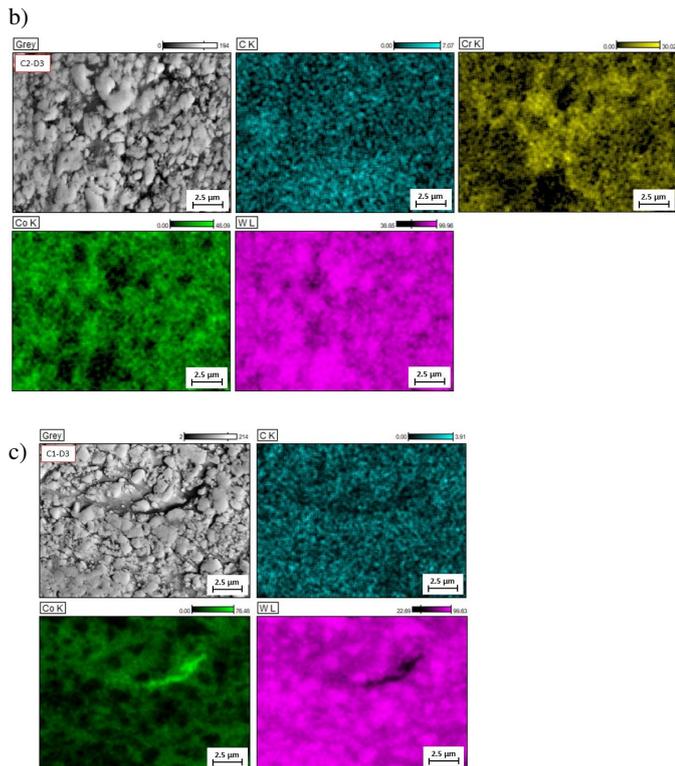


Fig. 3. The topography of the HVOF-sprayed coatings, (SEM)

Based on the microstructure investigations, it was found that the manufactured coatings are characterized by a dense and relatively uniform structure (Fig. 4a) which is characteristic for HVOF process [29–31]. Detailed examination revealed that the porosity level is low, and the pores are fine (much lower than  $1$   $\mu\text{m}$ ). Such fine pores could improve the corrosion resistance of sprayed coatings [32].





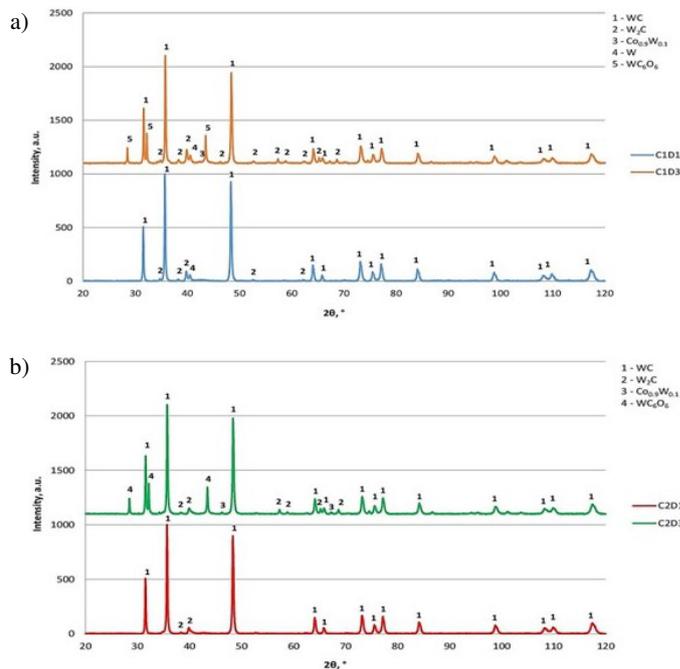
**Fig. 4.** The microstructure of the HVOF-sprayed coatings: a) middle area of the coating; b), c) elemental distribution maps of elements in the analyzed area, (SEM)

Additional analysis of the deposited coatings by EDS and elemental distribution maps confirmed that samples consist of WC particles embedded in a Co or Co–Cr matrix, depending on the used feedstock material. SEM images of the coatings showing unmelted carbide particles in the matrix (Figs. 4b and 4c). Furthermore, their shape clearly shows that they were partially melted during the HVOF spraying process. Similar results could be found in [33]. The average coating thickness ranges from 250 up to 350  $\mu\text{m}$ . On the other hand, there are some material discontinuities that could be seen, especially for longer spray distances, which do not allow us to form a dense structure. Also, the lower content of the cobalt matrix for C2 coatings could have an impact on increasing porosity values [34, 35].

### 3.2. Phase composition

The phase composition of the manufactured coatings consists of two hexagonal carbides: WC and  $\text{W}_2\text{C}$ , hexagonal Co, and cubic solid solution of tungsten in cobalt with composition  $\text{Co}_{0.9}\text{W}_{0.1}$  [36]. For the longest spray distance (samples C1–D3 and C2–D3) also a small quantity of  $\text{WC}_6\text{O}_6$  phase has been identified as a consequence of partial oxidation of WC (Fig. 5). Such results were confirmed by [37, 38]. For additional analysis of the phase composition, the index of carbide retention (ICR) was proposed [39]. The obtained results of ICR values for WC–Co samples were equal to 0.91 and 0.92 for C1–D1 and C1–D3, respectively. For WC–Co–Cr samples, ICR values were equal to 0.95 and 0.96 for C2–D1 and C2–D3, respectively. The presence of chromium reduces the tendency of  $\text{W}_2\text{C}$  formation as

well as the increasing spray distance [40]. It should be stressed that the ICR value closer to the unity is better in terms of coatings cohesion and wear- and corrosion resistance [41].



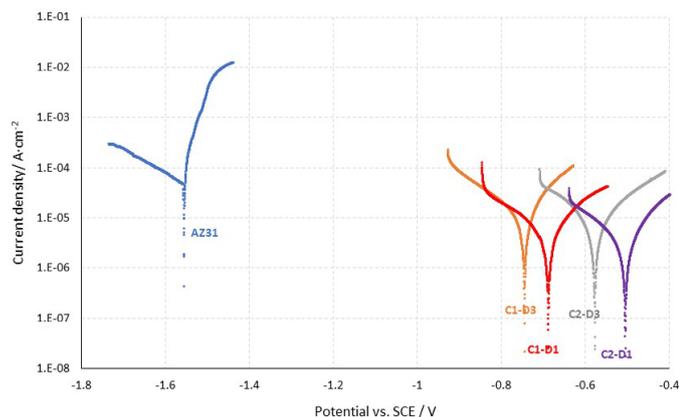
**Fig. 5.** The phase composition of the HVOF-sprayed coatings (SEM): WC–Co (a) and WC–Co–Cr (b)

### 3.3. Corrosion resistance

The results of the potentiodynamic investigations revealed that coated samples exhibit much better corrosion resistance than uncoated magnesium alloy AZ31 (Fig. 6) in the environment of sodium chloride solution. A detailed analysis of the potentiodynamic curve indicates that AZ31 alloy in the 3.5% NaCl solution started to oxidize. Such behaviour was observed also in other light material alloys, e.g. [42]. For coated samples, a decrease in the corrosion current density ( $j_{\text{corr}}$ ) and an increase in the corrosion potential ( $E_{\text{corr}}$ ) could be observed. The presence of the cermet coating inhibits anodic processes (more flat curve shape).

The polarization resistance of the magnesium alloy is very low ( $241.3 \Omega\text{-cm}^2$ ) whereas the corrosion current density is high ( $57.4 \mu\text{A}\text{-cm}^{-2}$ ). The corrosion potential of the AZ31 alloy equals  $-1.56 \text{ V}$ , which is characteristic of the magnesium alloy in NaCl solution [43–45]. The manufacturing of the protective coatings on the AZ31 alloy substrate by HVOF led to a significant shift of the corrosion potential towards more positive values. In the specified electrochemical system, all the coatings have a cathodic character and in the case of corrosion cell formation, the magnesium alloy substrate as an anode will corrode first. The biggest change of  $E_{\text{corr}}$  was registered for the samples C2–D1 and C2–D3 ( $-0.506 \text{ V}$  and  $-0.579 \text{ V}$ , respectively). Also samples C1–D1 and C1–D3 also result in a significant shift of the corrosion potential towards more positive values ( $-0.690 \text{ V}$  and  $-0.747 \text{ V}$ , respectively). Similar values were reported by Utu [46] and Shabana [47].

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**Fig. 6.** Potentiodynamic curves for the cermet coatings and uncoated AZ31 as a reference

Similar behaviour of the two types of coatings was noticed in relation to the spray distance. It was noticed that in the case of a shorter distance (320 mm), the corrosion potential values were higher than for the distance of 400 mm. The difference in  $E_{\text{corr}}$  values in the two types of coatings was similar, i.e. 74 mV (for C1 samples) and 57 mV (for C2 samples). In the case of the coatings formed by using the distance of 320 mm, the polarisation resistance ( $R_p$ ) values were higher (and corrosion current density values were lower) than for the coatings sprayed by using the distance of 400 mm. It could result from a more compact structure. Such a phenomenon was registered by Hong [48].

It should be stressed that all HVOF sprayed cermet coatings exhibit good protection properties for the magnesium alloy. Nevertheless, the best improvement of corrosion resistance was provided by the C2–D1 sample. This coating was characterized by c.a. 20 times lower corrosion current density ( $j_{\text{corr}} = 2.9 \mu\text{A}\cdot\text{cm}^{-2}$ ) in comparison to magnesium alloy substrate ( $j_{\text{corr}} = 57.4 \mu\text{A}\cdot\text{cm}^{-2}$ ). The other coatings have also lower corrosion current density (see Table 3). Similar results could be found in [47, 49, 50].

Additionally, in Table 3 it could be clearly seen that polarization resistance is also even 20 times higher for coated sample (C2–D1) in order to uncoat the AZ31 substrate.

**Table 3**

The results of the potentiodynamic measurements in 3.5% NaCl solution

Sample code	Corrosion current density ( $j_{\text{corr}}$ , $\mu\text{A}\cdot\text{cm}^{-2}$ )	Polarization resistance, $R_p$ ( $\Omega\cdot\text{cm}^2$ )	Corrosion potential ( $E_{\text{corr}}$ ), vs. SCE (V)
AZ31	57.4	241.3	-1.557
C1–D1	3.5	6733	-0.690
C1–D3	11.7	2621	-0.747
C2–D1	2.9	8423	-0.506
C2–D3	7.3	4180	-0.579

### 3.4. Roughness, porosity and microhardness of deposited coatings

The values of surface roughness and porosity are given in Table 4. The tendency is clearly observed, a shorter spray distance results in lower porosity of the coatings. On the other hand, the porosity depends on many technological parameters, for example, the type and shape of the feedstock powders [51, 52].

In the case of surface roughness, the slight influence of the spray distance could be observed, but the differences are in the range of standard deviation. Such values are typical for standard HVOF cermet coatings [53, 54].

Table 4 also includes the results of microhardness estimation. It was found that the highest value is exhibited by sample C1–D1, whereas the lowest one was characteristic for sample C2–D3. Such results revealed that a shorter spray distance promoted a more compact structure with a lower amount of pores [55, 56].

**Table 4**

Average values of roughness ( $R_a$ ,  $R_z$ ), porosity and microhardness of deposited coatings

Sample code	Roughness parameter ( $\mu\text{m}$ )		Porosity (vol.%)	Microhardness HV0.3
	$R_a$	$R_z$		
C1–D1	$3.81 \pm 0.27$	$22.25 \pm 1.86$	$1.9 \pm 0.5$	$1305 \pm 148$
C1–D3	$3.59 \pm 0.24$	$20.98 \pm 1.08$	$2.8 \pm 0.7$	$1085 \pm 176$
C2–D1	$4.08 \pm 0.24$	$23.30 \pm 0.87$	$2.3 \pm 0.5$	$1278 \pm 127$
C2–D3	$3.82 \pm 0.17$	$22.60 \pm 1.50$	$3.1 \pm 0.6$	$1042 \pm 186$

## 4. CONCLUSIONS

In this study, WC–Co and WC–Co–Cr coatings deposited by HVOF thermal spraying from commercially available feedstock powders onto AZ31 magnesium alloy with different spray distances (320 and 400 mm) were inspected in terms of their microstructure, phase composition and electrochemical corrosion resistance. The experimental results can be summarized as follows:

- All manufactured coatings revealed a topography and microstructure typical for the HVOF process, with regular and smooth surfaces as well as a compact structure. Moreover, in the coatings, the carbide distribution is even.
- Investigations of phase composition revealed the presence of hexagonal WC,  $\text{W}_2\text{C}$  and Co as well as a cubic solid solution of tungsten in cobalt ( $\text{Co}_{0.9}\text{W}_{0.1}$ ). Moreover, for longer spray distances, the small quantity of  $\text{WC}_6\text{O}_6$  phase was identified as a consequence of partial oxidation of WC.
- The clear influence of the spray distance on corrosion resistance was observed. The  $j_{\text{corr}}$  values were lower, whereas  $R_p$  values were higher for shorter spray distances, which improves corrosion resistance.
- The same influence of the spray distance was also observed in the case of porosity and microhardness. A lower porosity value was connected with better corrosion resistance.

## ACKNOWLEDGEMENTS

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