THE EFFECT OF SPRAY DISTANCE ON POROSITY, SURFACE ROUGHNESS AND MICROHARDNESS OF WC-10Co-4Cr COATINGS DEPOSITED BY HVOF

ABSTRACT

The paper presents the computational studies on the microstructure of WC-Co-Cr coatings deposited by High Velocity Oxy Fuel spraying (HVOF). The study covers the porosity assessment according to ASTM E2109-01 standard, carried out in ImageJ software, in terms of volume porosity, size and shape of the pores. The evaluation was preceded by scanning electron microscope (SEM) observations at magnifications of 2000x and 5000x. Additionally, topography analysis has been performed by confocal laser scanning microscope (CLSM), and the surface roughness Rₐ was evaluated by the contact method with use of a stylus profilometer. Finally, the influence of porosity was observed for coatings microhardness HV0.3. According to the results, the total closed porosity was found to be in the range of 5.01 vol.% and 5.38 vol.%. The dominated pores in the coatings were of size 0.1-1.0 µm. Studies showed that HVOF process enabled deposition of dense coatings, characterized by homogenous distribution of pores and low roughness.

Keywords: cermet coatings; High Velocity Oxy Fuel Spraying; porosity; computational image analysis

INTRODUCTION

Thermal spraying methods are widely used in industry due to the possibility of obtaining unique properties for a given process, impossible to achieve with conventional methods, the ease of automation, and a wide range of applications. Coatings applied by the high velocity oxy-fuel (HVOF) flame spraying method, thanks to very good adhesion, low porosity, and the possibility of using a different coating materials, allow to improve the corrosion-, erosion-, wear- and cavitation resistance of machine parts used in various industries [1-3]. The most important applications of the HVOF method include the spraying of cermet coatings based on tungsten carbide (WC), where, due to the low temperature achieved by the particles, the carbide transformation occurs to a small extent. Due to the fact that tungsten carbide (WC) can be well wetted, e.g. by cobalt (Co), nickel (Ni), iron (Fe), and cobalt - chromium (CoCr), cermet materials based on this carbide are one of the most commonly used cermet materials [4-6]. Moreover, there are also other materials deposited by HVOF method [7-9] and various
modifications, e.g. high velocity air-fuel (HVAF) [10, 11], suspension HVOF (S-HVOF) [12, 13].

Furthermore, important advantage of this kind of thermal spraying method is that it enables the appropriate combination of particle velocity and process temperature to generate dense coatings with porosity, which can be in the range from 0.5 to 5 vol. %, depending on the type of coating material, parameters of the spraying process and the spraying gun system [14] at high deposition efficiency and high powder feed rates [15]. The coatings obtained in this process can be used also (thanks to their high density, hardness, and toughness) in applications where fracture toughness is essential [16]. Many parameters affect the properties of the coatings. The most important factors are microhardness, porosity, roughness, and thickness [17]. Porosity is a key microstructural feature of thermal spray coatings. The pore and crack network originate during the chaotic processes of flattening and solidification of impacting molten droplets [18]. Porosity, such as a prevalent feature in the microstructure, affects a wide range of coating properties—among others, elastic modulus, thermal conductivity, and dielectric behavior [19, 20]. S. Paul [21] drew attention to the fact that the presence of a large number of defects, in the form of interlamellar pores and microcracks, are responsible for the low as-sprayed Young’s modulus and thermal conductivity of the top coats. In one side, reducing the amount of pores in dense thermally sprayed coatings resistant to abrasive wear, is important, because the presence of them can deteriorate the overall tribological performance of the coating [22]. Beyond the pores volume fraction, another important factors which determine properties of the coatings, are size and shape of the pores [23]. Additionally, a homogeneous pore size distribution are desired to achieve a high strength of the composite [24]. The microstructures of thermally sprayed coatings are very complex and incorporate process-dependent defects such as globular pores, interlamellar pores, cracks (in the case of ceramics), etc. [19]. Due to the brittle nature of ceramic materials, some intra-splat cracks (parallel to the heat flux) are formed inside layers during the process of thermal spraying. Another type of cracks are inter-splat ones, which are mostly perpendicular to the heat flux. It is reported that both types may occur in the coating after spraying [25].

When investigating the porosity, it should be considered features like pore size distribution, pore volume and the pore shape [19, 20]. However, there is no single method that can be adequately applied because of a very wide scale of porosity size. It can be started from a few nanometers and end with centimeters [26].

Porosity is a relatively easy parameter to define, but certainly not so easy to quantify [26]. The evaluation of coating porosity is a challenging task [27], although there are numerous methods for determining it [19-21,27]. For analyzing porosity, among others the techniques described below can be used: (i) Mercury intrusion porosimetry (MIP) - based on the assumption that non-wetting liquid will only intrude the pores under pressure and that pores have cylindrical geometry. It evaluates only open porosity but provides detailed information on pore size distribution; (ii) Water adsorption (WA), which is an inexpensive and single method, but does not measure closed porosity; (iii) Helium pycnometry (HP), which measures open porosity but does not provide the pore size distribution; (iv) 3D reconstruction phase contrast by X-ray microscope (3D XRM) measures both open and close pores, with the size of pores up to 50 nm, which is a new but expensive technique; (v) NMR cryoporometry- measures pores down to nanometer size, approximates pore shape, a new technique [27] and at finally (vi) Image analysis (IA) - this method detects both open and
close pores, allows to determine distribution of pore size; magnification and image contrast can influence the results. Due to the possibility of quick and simple conduct of the study with this method, it was used in the presented research. The accuracy of this method depends significantly on the metallographic preparation and metallography procedure for coating specimens [27, 28].

Manufacturing cermet coatings by HVOF should provide a dense structure with good adhesion. To ensure good resistance against wear, erosion, cavitation and corrosion, a low porosity level is required. Among many methods of porosity assessment, image analysis (IA) could be a promising technique, which combines two advantages: precision and relatively high performance.

In the present paper three different coatings deposited by high velocity oxy-fuel (HVOF) with different spray distances have been compared in terms of their porosity level, surface topography, and microhardness.

MATERIALS AND METHOD

Feedstock characterization and substrate preparation

A commercially available powder of tungsten carbide with the addition of chromium in cobalt matrix (WC-Co-Cr, Amperit 554.071 made by Höganäs) was used as a coating material. The chemical composition of WC-Co-Cr powder (in wt.%) was: 86, 10 and 4, respectively. Scanning electron microscopy (SEM) image presents morphology of the initial powder (Fig. 1). The particle size distribution declared by the manufacturer was confirmed and it was - 25 + 5 µm. As a substrate, a structural steel, S235JR (according to the EN 10027-1 standard), was used. The thickness of the plate was 4 mm. Before the deposition process, the surface of the substrates was grit-blasted with corundum F40 (according to the FEPA standard) to obtain larger specific surface area and, consequently, better coating adhesion to the substrate. After grit blasting and just before spraying, the substrates have been cleaned in the ultrasonic bath.

Fig. 1. Morphology of feedstock powder (SEM)
Deposition of the coatings

Manufacturing of the coatings was carried out using a C-CJS spray system (Thermico). The thermal spraying has been carried out in CERTECH Company (Wilamowice, Poland). Constant process parameters have been collected in Table 1.

<table>
<thead>
<tr>
<th>Table 1. Constant process parameters</th>
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<tbody>
<tr>
<td>Fuel flow rate, l/min</td>
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<tr>
<td>Oxygen flow rate, l/min</td>
</tr>
<tr>
<td>Gas flow rate, l/min</td>
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<tr>
<td>Powder feed rate, g/min</td>
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<tr>
<td>Gun velocity, mm/s</td>
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</table>

On the other hand, the variable process parameters, as well as, the sample code have been presented in Table 2. Such values of spray distance resulted from recommendations of the powder manufacturer and also from company experience with such material as well as from work with above mentioned set-up.

<table>
<thead>
<tr>
<th>Table 2. Sample code and variable process parameters</th>
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<tbody>
<tr>
<td>Sample code</td>
</tr>
<tr>
<td>A</td>
</tr>
<tr>
<td>B</td>
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<tr>
<td>C</td>
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Porosity analysis

Porosity analysis was carried out using ImageJ open source software. For each sample, 15 images were taken at magnification of 2000x and 5000x. These images have been carried out by Scanning Electron Microscope Tescan Vega 3 (Tescan Orsay Holding). The goal of the porosity investigation was to determine the volume of pores, depending on the average pore size and its circularity [29]. In total, three different size ranges were distinguished: (i) 0.01-0.1 µm, (ii) 0.1-1 µm and (iii) > 1 µm. Circularity was divided in the ranges: (i) 0-0.25 (which stands for the most irregular pores), (ii) 0.26-0.50, (iii) 0.51-0.75 and (iv) 0.76-1.00 (where 1 means the perfect sphere shape). More details of porosity analysis methodology were presented in works [27, 30, 31].

Microhardness and topography

Topography analysis as well as the surface roughness of the obtained coatings have been carried out by non-contact Confocal Laser Scanning Microscopy (CLSM) of LSMS Excite Zeiss. The measurements have been carried out according to the ISO 13565-2 standard. In order to confirm the values of surface roughness, the stylus profilometer (MarSurf PS 10) has been used. The measurements have been carried out according to the ISO 4288 standard. The microhardness of the coatings was measured with Vickers penetrator under the load of 2.94 N.
RESULTS AND DISCUSSION

Microstructure of the coatings

The microstructures of the manufactured coatings are collected in Figure 2. As it could be seen, all coatings are well adhered to the substrate and the coating material filled the surface roughness. All coatings are characterized by relatively dense and homogeneous structure. Such type of structure is typical for HVOF coatings [32-35]. Moreover, the thickness of the deposited coatings was c.a. 100 µm.

![Image](image_url)

Fig. 2. Cross section of manufactured coatings: a) – sample A, b) – sample B, c) – sample C

The surface roughness of the coatings depends on the process parameters – an increased roughness was observed with increased spraying distance. In general, a relatively smooth surface of as-sprayed HVOF coatings could be achieved. Nevertheless, in the flame only the matrix is melted. The hard and high melting point carbide particles are not dissolved [36]. The surface topographies obtained by CLSM are collected in Figure 3. Surface roughness (R_a - the arithmetic mean of ordinates of the roughness profile) results are collected in Table 3. These results good correspond with the literature [37]. Results of surface roughness obtained from stylus profilometer confirmed the values from CLSM measurements and also corresponded with typical data of HVOF coatings in the references [38, 39].

<table>
<thead>
<tr>
<th>Sample code</th>
<th>R_a, µm (by CLSM)</th>
<th>R_a, µm (by stylus)</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>4.0 ± 0.5</td>
<td>4.2 ± 0.8</td>
</tr>
<tr>
<td>B</td>
<td>4.2 ± 0.7</td>
<td>4.4 ± 0.9</td>
</tr>
<tr>
<td>C</td>
<td>4.7 ± 0.1</td>
<td>5.1 ± 0.3</td>
</tr>
</tbody>
</table>
Fig. 3. Surface topography and surface roughness (Ra) of manufactured coatings:

a) – sample A, b) – sample B, c) – sample C
Porosity evaluation

The volumetric porosity level results obtained by IA have been estimated based on SEM images under magnifications equal to 2000x and 5000x. Exemplary images at higher magnification are collected in Figure 4.

Fig. 4. Cross section of manufactured coatings at 5000x magnification:
   a) – sample A, b) – sample B, c) – sample C

According to the ASTM E2109-01 standard and the methodology described in [27], the volumetric porosity levels have been estimated. The scheme of porosity assessment methodology is presented in Figure 5.

Fig. 5. Scheme of porosity assessment in HVOF sprayed coatings

The results are collected in Figure 6. As it could be seen, the total volumetric porosity is completely independent of magnification. Moreover, the spray distance (varying in current investigations from 240 up to 320 mm) was also without influence on the porosity level. All values are in the range from 5 up to 5.5 vol. %. In general, for HVOF coatings such results are relatively high. On the other hand, the initial feedstock was sintered, not agglomerated, which is a significant difference. The agglomerated powder creates splats with better interlamellar cohesion and with reduced porosity [40].
Fig. 6. Average porosity of deposited coatings

Requirements of ASTM E2109-01 standard for relative accuracy gives the information about the precision of image analysis for porosity estimation. This accuracy should be lower than 10%. For all investigated coatings and, what is essential, all ranges of pore size, this parameter has a value below 10%.

For all deposited coatings, the computed porosity volume fraction showed similar tendency. Mainly the pores are in the range from 0.1 up to 1.0 µm. All three ranges of pore size could be distinguished only at higher magnification (5000x). The results of pore size analysis and assignment to the proper range are presented in Figure 6. One of the possible explanation could be the size of initial powder particles (between 5 and 25 microns).

Detail analysis of Figure 7 gives the information that the lower fraction of pore size, below 0.1 µm is mainly characterized for the sample A (the shortest spray distance). It means, that the structure of the deposited coating is the most compact. On the other hand, the total volume porosity for this sample is very similar to the other one.

Fig. 7. The total volume fraction of pores computed for different pore sizes (for all coatings at mag. 5000x)
Apart from the size of the pores also the circularity has been calculated. This parameter determines the globularity of the pores. Details of the methodology and calculations could be found in [27]. The circularity of the pores in the manufactured coatings are collected in Figures 8 and 9 for 2000 and 5000 magnifications, respectively.

![Graph](image1.png)

**Fig. 8.** The total volume fraction of pores computed for different pore shapes (for all coatings at mag. 2000x)

![Graph](image2.png)

**Fig. 9.** The total volume fraction of pores computed for different pore shapes (for all coatings at mag. 5000x)

Figures 8 and 9 should be analyzed together. As it could be seen, for both cases the most dominant fraction is the most globular shape of the pores. Nevertheless, this fraction is even 15% lower for higher magnification. On the other hand, the fraction of average circularity (in two groups: 0.26-0.50 and 0.51-0.75) is higher about 15% for magnification 5000x. For both cases, the smallest circularity, which means elongated polygons, are at the same, very low level (below 2%). This result could well correspond to Fig. 4. For pore shape, the relative accuracy value is also below 10%, which confirms the precision of this method.

The influence of size of the pores and spraying distance could be observed for microhardness values (Fig. 10). The shortest spray distance exhibits the highest
microhardness. The total porosity is almost equal, but for sample A, the smallest fraction of the pores is the highest of all coatings. It could be the explanation of this phenomenon. On the other hand, similar results of the microhardness could be found in [5].

![Graph showing microhardness of coatings A, B, and C]

**Fig. 10.** Average microhardness (HV0.3) of manufactured coatings

**SUMMARY**

The porosity assessment of thermal spray coatings requires good techniques and has to be carried out with a lot of attention to details. It is much more important for coatings manufactured by methods, which are characterized by dense structures and low porosity level, like HVOF or cold spray. As it was presented in this paper, image analysis could be a reliable tool for porosity estimation. There are some advantages, like the possibility of measuring open and closed porosity, there is no need of advanced set-up required, repetitive results. On the other hand, there are some disadvantages, firstly it is time consuming (analysis of each image) and strongly depends on image quality. Additionally, magnification is one of the key parameters.

In the present investigation, image analysis confirmed that size of the pores mainly depends on the initial powder particle size (and its distribution), but it is not strongly influenced by the spraying distance. On the other hand, the value of volume porosity in total mainly depends on the powder delivery conditions (it is lower for agglomerated and sintered than sintered only). And finally, microhardness measurements confirmed the dependency on spray distance, the shorter distance exhibits higher microhardness.

**REFERENCES**


