



Influence of HVOF deposition thickness on adhesion strength of WC–CrC–Ni coatings

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ABSTRACT

In this paper, adhesion strength of WC–CrC–Ni coatings obtained by HVOF thermal spraying process of WOKA7504 powder has been studied in correlation with deposition thickness, chemical and structural properties. Besides the parameters strictly related to the process, deposition thickness was found to be the most significant factor affecting the adhesion strength. In the frame of experimental program 304L stainless steel and S235JR structural steel were used as substrate. Mechanical tests, SEM and EDX analysis, have been performed in order to assess structural and mechanical properties, phase composition of the deposited layers. Maximum average adhesion strength has been experimentally found of 23.4 N/mm² for stainless steel and of 35.8 N/mm² for structural steel, for a deposition thickness of 220 μm, achieved in two layers. Once the coatings' thickness increases, the adhesion strength decreases constantly and over thickness of 400 to 650 μm, the existing coating imperfections and elongated carbides placed in the interlayer interface area could fail due to the thermal stresses during the HVOF thermal spraying process, since they are brittle stress concentrators and thus leading to cracking of coatings at small loads, since the coating layers have high hardness of 990 to 1151 HV 1.

Key words: *protective coatings, tungsten carbide, HVOF, adhesive strength, failure mechanism*

1. INTRODUCTION

This Currently thermal-sprayed coatings are largely used to increase the lifespan and service performance of components. Other applications include component repairing and fabrication of net-shape components [1] and general fabrication of components protected by wear resistant layers [2]-[7], by erosion - corrosion resistant layers in aggressive environments [8]-[12] or by layers used as thermal barrier [13]-[16].

High Velocity Oxygen Fuel (HVOF), thermal spraying [17] has been a very successful process in the spraying of wear resistant coatings due to superior adhesion strengths, higher density and less decarburization, relative to many other thermal spraying processes. The WC–CrC–Ni coating layers, achieved by the HVOF process, have very high hardness and good corrosion resistance and thus can be used to protect different components (e.g. pipes, elbows, vane pumps, cylinders, valves, mining equipment etc.) and are used in various industries, as an alternative to hard chrome plating. The process parameters used to obtain the coating have a major effect on the maximum temperature of components, reached during the process,

resulting in a significant difference in the microstructural properties [18]-[20] and mechanical behaviour of the coatings [21], [22], residual stress after deposition [23], which affect the component performance.

Study on tribological and corrosion behaviour [24], [25], and on grinding effects on the adhesion strength [26] of HVOF thermally sprayed WC composite has been done before, but the potential of this type of coatings has not yet been exhausted. Due to the complexity of the process (large number of technological parameters, e.g.: spraying distance, spraying gun travel speed, oxygen-fuel ratio and deposition temperature, type and flow ratio of powder, particle size distribution and its production method, chemical composition of the metallic binder, mechanical properties of substrate, including surface roughness and oxidation state, post-treatment etc.), [27]-[29], more intensive studies are required for process optimisation, [30] and to define correlations between process parameters, coating composition and the microstructure obtained [31], [32], physical and mechanical properties of coatings, including bonding interface properties [33], coating thickness and the achieved coating adherence strength. Different qualitative or quantitative methods

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allow assessing the adhesion strength of thermally sprayed coatings [34]-[36], e.g. scratching test, tensile test, shear test, LASer adhesion test, bulge and blister test, interfacial indentation test and three-point bend test. In this paper, the influence of deposition thickness on adhesion strength of WC–CrC–Ni coatings obtained by the HVOF thermal spraying process was investigated. The EN 582 standard [37] was used to perform the adhesion tests.

2. EXPERIMENTAL WORK

2.1 Material and methods

The tests were performed on S235JR carbon steel and 304L stainless steel blocks used as substrate, protected by WC–CrC–Ni coating layers obtained by HVOF process using WOKA 7504 powder. The chemical composition and the mechanical properties of the substrates are presented in table 1 and table 2, respectively. The chemical composition and characteristics of the powder are presented in table 3 and table 4, respectively. The method used to assess the adhesion strength of WC–CrC–Ni coatings was according to the EN 582 standard [37].

Table 1. Chemical composition [%] of substrate materials [38], [39].

Steel	C max	Cr	Ni	Si max.	Mn max.	P max.	S max.	N max.	Cu max.
304L (1.4307)	0.03	18.0-20.0	8.0-12.0	1.0	2.0	0.045	0.03	-	-
S235JR (1.0038)	0.19	-	-	-	1.5	0.045	0.04	0.01	0.60

Table 2. Mechanical properties of substrate materials [38], [39]

Steel	R _m [N/mm ²]	R _{p0.2} [N/mm ²]	A _{min} [%]
304L (1.4307)	min.515	min.205	4
S235JR (1.0038)	360-510	min. 225	2

Table 3. Chemical composition [%] of the powder WOKA 7504, manufacturer's data [40]

Powder	Cr	Ni	Co	C	Fe	W
WOKA 7504	38.5 – 43.5	10.0 – 13.0	2.9 – 4.1	7.7 – 8.5	< 0.5	the rest

Table 4. Characteristics of the powder WOKA 7504, manufacturer's data [40]

Classification	Cr and W carbides
Chemical composition	Cr ₃ C ₂ 37WC 18Metal Alloy
Manufacturing method	Agglomeration and sintering
Morphology	Spheroidal
Dimensions	10 to 30 μm
The aspect	Rough
Apparent density	3.1 – 3.8 g/cm ³
Operating temperature	< 700 °C
Role	Corrosion and wear resistant
Recommended process	HVOF

2.2 Sample preparation and experimental setup

In order to perform the tests, special samples with a diameter of 25 mm were made of S235JR and 304L steel,

according to the above-mentioned test standard. The sample consists of two identical cylindrical blocks. On the front surface of each block, a protective coating layer of WC–CrC–Ni was deposited by HVOF thermal spraying process. Before performing the hard layers' deposition, both types of materials used as substrate have undergone a process of preparation by degreasing (with alcohol) and sandblasting of surfaces (with Al₂O₃ particles size F20 of 850-1180 μm, having high hardness and rugged shapes), mandatory procedure to be done in order to obtain proper quality surfaces for high performance coatings. This procedure is used not only for rust removal to get a virgin surface, but also to provide a compressive stress on the substrate surface and to generate anchor pattern texture, which results in appropriate mechanical bond characteristics of the coating to the substrate. Thermal spraying of coating was performed right after completion of the surfaces preparation in order to avoid forming of a thick oxide layer on the components' surfaces, which could affect the deposition quality. In order to obtain coatings with controlled thickness, an experimental setup has been designed, consisting of specialized HVOF (High Velocity Oxygen Fuel) thermal spraying equipment, which has a control module, a spraying gun mounted on a robotic arm and a powder feeder. Since the layer thickness depends on many parameters of the deposition process, during experiments some of process parameters remained constant: air flow 200 l/min, oxygen flow 185 l/min, propane flow 68 l/min, spraying gun – piece offset 225 mm, the powder flow 60 g/min, the spraying gun travel speed 75 mm/s and the step between two successive passes was calculated, based on the deposited material distribution by spraying in the given conditions.

Based on the distribution shown in Fig. 1, the step between two successive passes was graphically determined, Fig. 2, in order to obtain a coating thickness as uniform as possible, Fig.3 (for one pass) and Fig. 4 (for two passes).

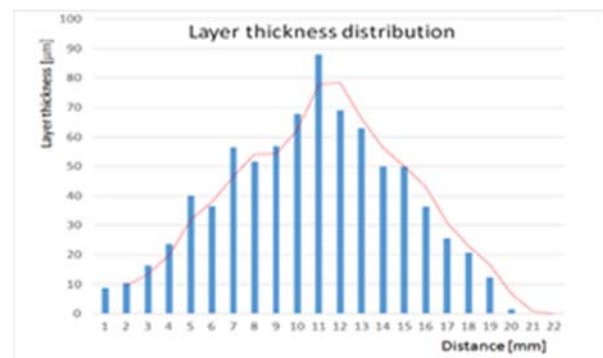


Fig. 1. Layer thickness distribution per pass

Thus, for the parameters above specified, the step used was $p = 9$ mm. Although the jet of sprayed particles comes into contact with the metal substrate for a very short time (value that varies depending on the parameters of HVOF thermal spraying process), however, the absorbed heat is accumulated in the substrate and, in the case of small components, it can reach very high values,

leading to component degradation or affecting the deposition quality. For monitoring the sample temperature during the HVOF process, an infrared camera FLIR System A40 and a dedicated software for recording of thermographic imaging were used.

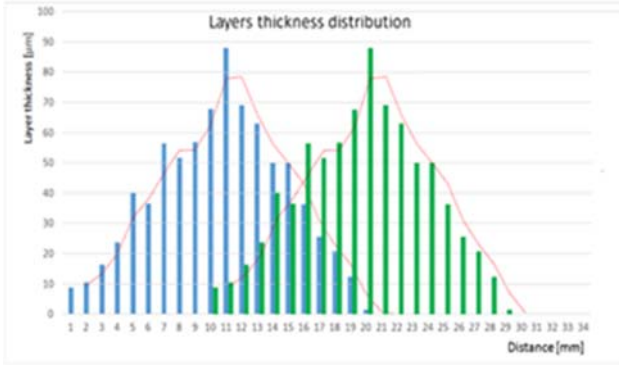


Fig. 2. Determining the distance between two passes

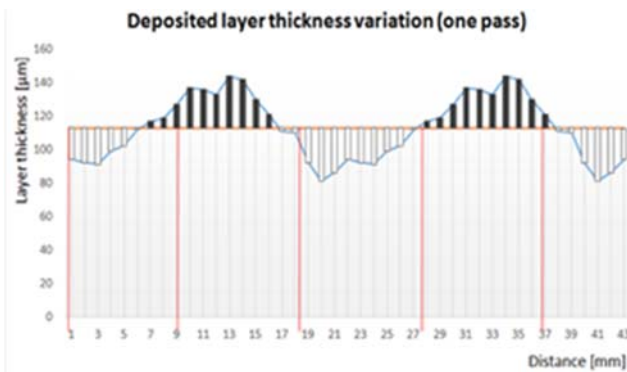


Fig. 3. Layer thickness – one pass

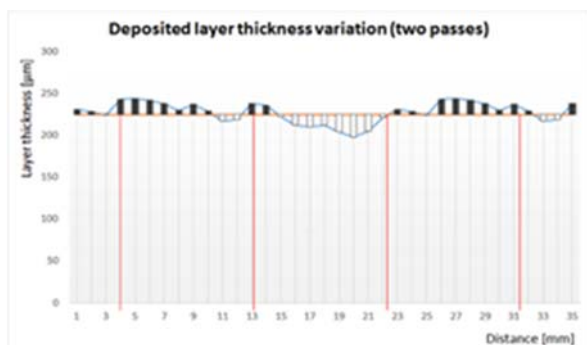


Fig. 4. Layer thickness – two passes

In order to assess the temperature distribution of the samples surfaces during the HVOF thermal spraying process, a plate of 200 x 200 mm was used as support and 12 passes were deposited on it. The same parameters were used as in the case of cylindrical blocks. Temperatures were recorded and the variation of the sample's surface temperature, during HVOF thermal spraying process, is presented in Fig. 5.

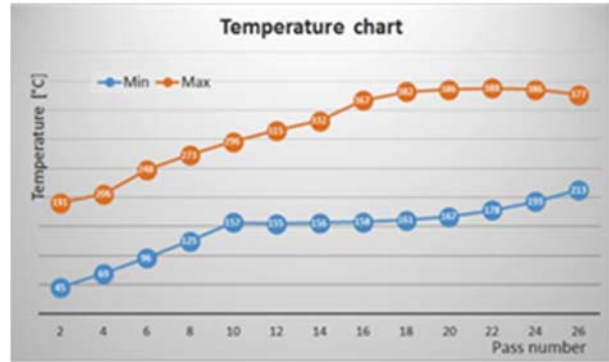


Fig. 5. Variation of the sample's surface temperature during HVOF process

It has been found that during the spraying process the sample temperature increases according to the number of passes and the number of layers deposited. Thus, although after completion of the first deposited layer (after 12 passes), 36% of the sample surface has a temperature of 291 to 325 °C and 64% of the sample surface has a temperature below 290 °C, in case of continuously deposition of two layers, at the end of the second layer, 25% of the sample surface has a temperature of 361 to 388 °C and only 14% of the sample surface has a temperature below 290 °C. After completion of the successive deposition layers, the cylindrical blocks are bound together using a high strength adhesive, so that the coated front faces come into contact. After making the adhesive bond, and waiting to solidify (24 hours), the obtained sample is mounted in the testing machine and tested for tensile strength. The loading rate was 1 mm / min and tests were made on three samples for each coating thickness, according to the number of layers deposited. The experimental program consists of two, four and six successive deposition layers on the two types of metal substrate, using HVOF thermal spraying process of WOKA 7504 powder. For each number of layers and each metal substrate, three samples were prepared and tested. Additionally, in order to assess the adhesive strength, three samples without deposited layers were prepared and tested. The testing equipment used was a tensile testing machine, EDZ 40 type, upgraded using Zwick / Roell technology, with a maximum load of 400 kN, equipped with grips and special devices for this type of test. Samples are clamped by the jaws of the tensile testing machine by means of threaded rods on semi-spherical recesses, so as to ensure sample centring and gripping without inducing lateral forces.

Micro hardness measurements were performed on the crosssection of the coatings using a Zwick 3212 equipment and a load of 1000 g for 10 s. SEM and EDX analyses were done with a Quanta FEG 250 Microscope, produced by FEI, equipped with an EDAX analyser and backscatter electron detectors that allowed to perform scanning electron microscopy analyses in BSD mode.

3. RESULTS AND DISCUSSION

From thickness measurements of deposited layers conducted in the experimental program, it appears that using the HVOF process parameters presented in section 2.2, a compact deposited layer (Fig. 6) with only few small pores, a good penetration of the coating into the substrate and a thickness about of 100 to 115 μm can be achieved.

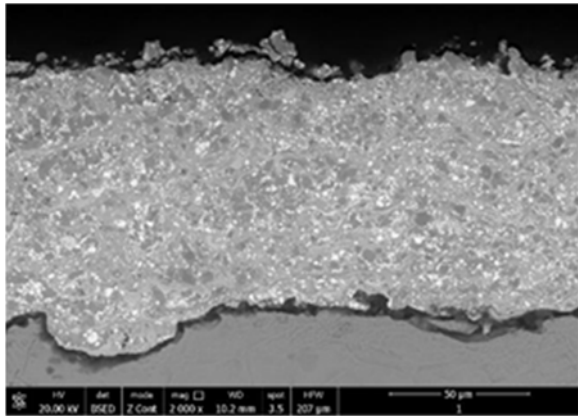


Fig. 6. The SEM microscopic aspect in cross-section of a sample with one deposited layer achieved by HVOF thermal spraying process of WOKA 7504 powder

The differences in the substrate roughness and deposited layers does not significantly influence the amount of metal deposited, thus the layer average thickness has a linear variation with the number of layers deposited, as is presented in Fig. 7.

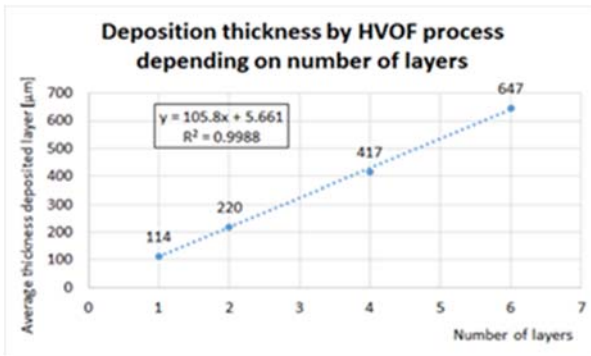


Fig. 7. Deposition thicknesses versus number of layers deposited by HVOF thermal spray process

In correlation with the deposition thickness, table 5 presents the results of the hardness tests on WC–CrC–Ni coatings. Are shown that the average hardness is high comparatively with the substrate (148 to 216 HV1) and slightly increases with the number of coating layers, except thick deposits of 6 layers where imperfections affect the hardness tests results as well.

Table 5. HVOF WC–CrC–Ni coatings - Hardness test results, HV 1, average values

1 layer	2 layers	4 layers	6 layers
1003	1099	1151	990

Table 6. Adhesion strength of WC–CrC–Ni coatings obtained by HVOF process, RH [N/mm²], average values

RH [N/mm ²]	2 layers	4 layers	6 layers
304L (1.4307)	23.4	11.7	8.0
S235JR (1.0038)	35.8	19.7	16.0
Epoxy Adhesive (BISON Metal)	37.5		

Table 6 presents the average value of adhesion strength of WC–CrC–Ni coatings obtained by HVOF process for two metal substrates. It was revealed that the adhesion strength varies depending on the metal substrate and with the number of layers deposited and thus on the total deposition thickness. To explain these variations, the values obtained for adhesion strength should be correlated with the macroscopic features of the samples' fracture surfaces revealed after performing the tests. Figure 8 presents the aspects of the fracture surface, resulting after the adhesion tests of sample with 2 coating layers, obtained by HVOF deposition of WOKA 7504 powder on S235JR substrate (Fig. 8a) and on 304L substrate (Fig. 8b).

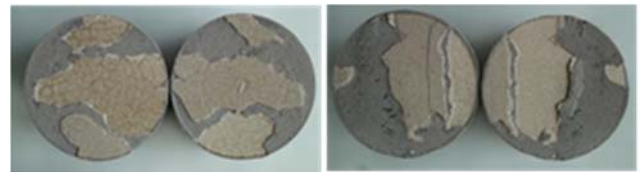


Fig.8. The aspect of samples fracture surfaces, with 2 deposited layers achieved by HVOF thermal spraying process of WOKA 7504 powder, after performing adhesion tests: a) 25 mm sample, S235JR substrate; b) 25 mm sample, 304L substrate

In both cases, the separation surfaces show a mixed fracture aspect. Failures occurred as well as on adhesive (dark grey) and on the interface between the deposited layers and the substrate. Macroscopically no cracks or tears between layers were detected. This aspect, together with the average value of adhesion strength of 35.8 N/mm² recorded in the case of S235JR substrate and of 23.4 N/mm² for 304L substrate, points out that both the adhesion of deposited layer of the two metal substrates and adhesion between the deposited layers are very good (closest to the adhesion strength of 37.5 N/mm² of the adhesive used in the process of samples preparation). The differences between the values of adhesion strength coming from the higher percentage of failure at the layer-substrate level for approx. 40% of total fracture surface in the case of 304L substrate, and less than 25% of total fracture surface for S235JR substrate.

Figure 9 presents the microscopic aspect of a sample with two deposited layer achieved by HVOF thermal spraying process of WOKA 7504 powder in the areas where the EDX analyses were made. Microstructural analysis reveal that W and Cr carbides are evenly distributed in the matrix and small amount of oxides are present, which shows that the process parameters were appropriate selected. It is highlighted that unlike the carbides

embedded in the matrix, which have a globular shape, the carbides placed in the interface area are elongated. As a result of their elongated shape, these carbides are stress concentrators and in combination with their high hardness can cause the layer failure by mechanical stress. The local chemical analyses using EDX technique were performed at the interlayer interface as well as on different constituents. It is highlighted that in the lighter areas the W is predominant, and in the dark grey areas the concentration of Cr is higher as well as C, Fe, Ni and Co are present in the matrix. The aspect of fracture surfaces for the samples prepared by depositing of 4 successive layers by HVOF thermal spraying process of WOKA 7504 powder is presented in Fig. 10. For the sample with the S235JR substrate, although macroscopically no cracks were revealed in the layer, failure occurred at the layer - substrate interface, Fig. 10a.

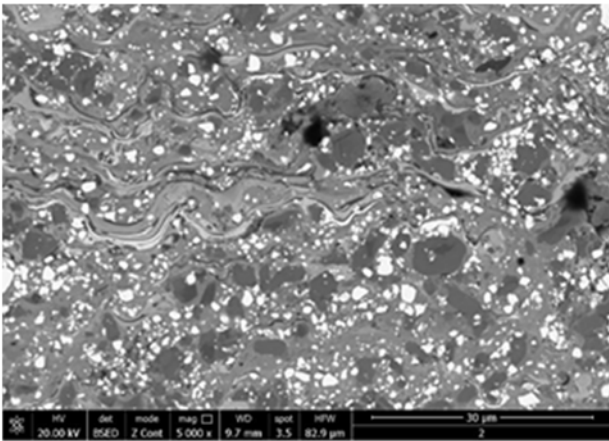


Fig. 9. The SEM microscopic aspect in cross-section of a sample with two deposited layer achieved by HVOF thermal spraying process of WOKA 7504 powder



Fig.10. The aspect of samples fracture surfaces, with 4 deposited layers achieved by HVOF thermal spraying process of WOKA 7504 powder, after performing adhesion tests: a) \varnothing 25 mm sample, S235JR substrate; b) \varnothing 25 mm sample, 304L substrate

This aspect, together with the average value of adhesion strength of 19.7 N/mm² emphasizes that, in this case, the layer adherence to the substrate is relatively low.

A lower adhesion strength of 11.7 N / mm² was recorded for the 304L substrate (about two times lower than the adhesion strength obtained in case of two layers), and macroscopically mixed fractures and cracks in the deposited layers were revealed, Fig. 10b. Figure 11 presents the aspects of the fracture surface resulting after the adhesion tests in case of 6 layers' deposition, after performing adhesion tests. In both cases (S235JR substrate, Fig. 11a and 304L substrate, Fig. 11b) the

failure surface shows a mixed fracture, at the layer - substrate interface and as well as between layers deposited successively.



Fig.11. The aspect of samples fracture surfaces, with 6 deposited layers achieved by HVOF thermal spraying process of WOKA 7504 powder, after performing adhesion tests: a) \varnothing 25 mm sample, S235JR substrate; b) \varnothing 25 mm sample, 304L substrate

Macroscopically, cracks in the deposited layer and local layer exfoliation were highlighted. These facts, in conjunction with lower average values of the adhesion strength of 16.0 N/mm² for S235JR support and only of 8.0 N/mm² in the case of 304L substrate (about 3 times lower than the adhesion strength obtained by depositing of two layers), show that the layer adhesion to the substrate is particularly low in these cases. Also, in the case of the S235JR substrate, a more intense colouring of the metal substrate can be noticed, that indicates a pronounced heating of the sample during the HVOF thermal spraying process occurred.

Figure 12 and 13 presents the micro structural aspect of a sample with six deposited layer, indicates the propagation direction of interlayer crack and reveals some fine cracks developed perpendicular to it toward the outer surface of the deposition (Fig. 12). The cracks were initiated in the inter layer interface area (Fig. 13) with elongated and hard carbides of W, that failed under thermal stress, and propagated through the matrix.

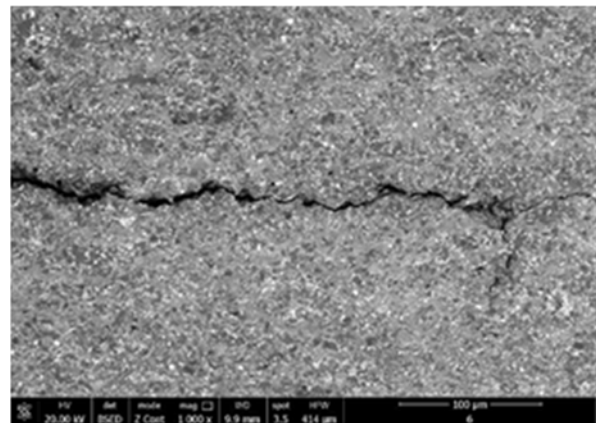


Fig. 12. The SEM microscopic aspect in cross-section of a sample with six deposited layer achieved by HVOF thermal spraying process of WOKA 7504 powder: inter and intralayer crack propagation

The properties of WC-CrC-Ni coatings obtained using optimal technological parameters of the HVOF process were previously investigated by W. Fang and collaborator [41] and by M. M. El Rayes and collaborator [42]. They found that these HVOF coatings are compact and are very protective for the substrate, having good thermal stability,

good corrosion resistance and excellent wear resistance. On the other hand, they concluded that the chemical composition of the metallic binder and the occurrence of micro cracks were the most important factors influencing the corrosion resistance of the HVOF sprayed WC coatings in the strong acidic environment. The enhanced corrosion resistance of HVOF coatings has been explained by S. M. Hassani-Gangaraj and collaborator [29], due to the presence of Cr₂O₃ in protective layers. Thereby, Chromium having very high affinity for oxygen, forms stable oxide and inhibits the substrate oxidation by blocking the diffusion of reacting species. Thus, the HVOF thermal spraying process is recommended to be used in the case where the oxides' formation is the main protective mechanism.

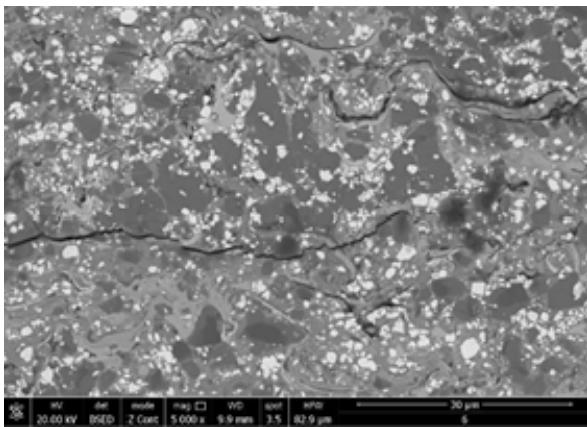


Fig.13. The SEM microscopic aspect in cross-section of a sample with six deposited layer achieved by HVOF thermal spraying process of WOKA 7504 powder: crack in the interlayer interface

The experimental results support these observations, allowing validation of assumptions that in case of WC-CrNi hard coatings, obtained by the HVOF thermal spraying process, the deposition thickness plays a decisive role on adhesion strength and on the existence of a critical thickness starting from which the deposition quality is affected. Additionally, it was also revealed that this critical value is closely related to the occurrence of pores and micro-cracks in the coating layers. This issue is linked to the technological parameters of the HVOF thermal spraying process, whereas by variation of the velocity and

temperature of the sprayed particle, the substrate temperature could be modified in order to avoid internal stress in the coating layers and finally to achieve the optimal deposited layer thickness.

Many experimental and analytical efforts have been carried out in order to provide a better understanding of the bonding mechanism in the HVOF thermal spraying process, but currently the accurate bonding mechanism is still a matter of debate. In general, a critical velocity for the spraying particle is needed to produce localisation of those to the substrate interface. As a result of particle impact, a strong pressure is exerted on the substrate and a shear load is generated, which accelerates the material laterally, and thus during high strain rate deformation,

strengthening occurs as a result of substrate metal dislocation and contraction of the plasticised sprayed particle. Usually increasing the temperature of the sprayed particle by using a proper oxygen–fuel ratio is good, since plastic deformation of the powders is induced and thus less porous coatings are obtained. On the other hand, in the case of continuous deposition of many layers, our experimental results reveal the fact that too much heat input into substrate is not good from the coating adhesion strength point of view, since the main adhesion mechanism is based on the mechanical anchoring of hot sprayed particles to the surface irregularities of the substrate, which occurs as a result of the sudden contraction of the plasticized particle in contact with the colder substrate, due to temperature differences between them. If the substrate temperature is too high, this effect does not occur and moreover cracks are initiated in or between the coating layers due to the internal stresses induced during cooling as a result of the difference between the thermal expansion coefficients of the substrate and coating materials.

4. CONCLUSIONS

The paper illustrates the influence of deposition thickness on adhesion strength of WC–CrC–Ni coatings obtained by the HVOF thermal spraying process of WOKA 7504 powder. In this case, the existence of a critical coating thickness, starting from which the adhesion strength, and generally the deposition quality, is affected due to the occurrence of elongated carbides or fine micro crack in the coating layer has been revealed. Best experimental results regarding the adhesion strength have been obtained for two layers deposited with a total thickness of 220 μm . Once the total thickness of the coating increased to over 400 - 650 μm , elongated carbides placed in the interlayer interface could fails due to the internal thermal stress induce by the HVOF process and micro crack occurred, which leads to the reduction of the adhesion strength and even causes layer cracking. It has been shown that the adhesion of WC–CrC–Ni thermal sprayed powder on the S235JR structural steel substrate is higher to that achieved for 304L austenitic steel substrate, due to a better mechanical anchoring of the plasticized particulate on the structural steel substrate. In this regard, tensile adhesion tests have shown an average adhesion strength of 35.8 N/mm² for structural steel substrate and of 23.4 N/mm² for stainless steel substrate, used in the experimental work. Whereas the technological parameters are closely related to the adhesion mechanism, by choosing the proper ones related to the heat input and to the substrate temperature, the HVOF process could be controlled in order to obtain high quality coatings. In the industrial applications, in order to reduce the internal thermal stresses due to overheating, and thus to reduce the risk of cracks occurrence in the coating layer of the component subjected to the HVOF thermal spraying process, the parameters of the process have to properly adapt to the shapes and dimensions of components. The

manufacturing of thick layers over 100 μm per pass should be avoided, since it significantly increases the substrate temperature and thus the residual stresses; (e.g. 30-50 μm per pass could be a good choice). The process must be carried out intermittently, so that the component can be able to cool before the deposition of the next layer. Monitoring the temperature of components is recommended to be carried out during and / or after the deposition of each layer.

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