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Technology and Characterization of Composite Thermal Spray Powders

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This article focuses on the characterization of WC-based composite spray powders produced by mechanically activated synthesis. The main characteristics of the powder (particle/granules size, size distribution, morphology, chemical composition and granules microstructure) were determined. Chemical composition of powder particles by energy-dispersive X-ray mapping technology and atomic emission spectroscopy was studied. For particle morphology image analysing method was used. Granularity of the produced powders was analysed by laser diffraction analyser. Also sieve and image analyses were used. The final goal was to obtain abrasive wear resistant coatings by thermal spray and to study their wear resistance. A good wear resistance of coating was demonstrated.

Keywords: Composite powders, nanostructured agglomerates, granularity and morphology, powder chemical composition.

1. INTRODUCTION

Thermal sprayed coatings are widely used in many industrial sectors to enhance the mechanical performance of components, and to strengthen and repair components that have been damaged in service. Moreover, a new coating must be more wear resistant and make components work proof.

Using commercially produced powders and high-velocity oxy-fuel (HVOF) process, high-density coatings (porosity less than 1 – 2 %) could be achieved. The coatings' properties do not depend only on the spray system but also on the spray powder characteristics. Earlier studies have demonstrated [1, 2] that the use of recycled hardmetal powder in the formation of detonation coatings leads to many problems. Hardmetal powder particles of sizes in the range from 32 to 40 μm, used for detonation spray, produce very porous (4 – 5 %) and not uniform coatings. Improvements of technological properties of spray powders from recycled hardmetal powder by different methods were carried out, but the desired coatings properties were not achieved [3]. The abrasion-erosion wear resistance of detonation sprayed hardmetal coating was low due to the peculiarities of the powders produced by impact milling from recycled hardmetal.

This paper focuses on the characterization of experimental WC-based composite spray powders produced by mechanically activated synthesis. The composite powder particle size distribution, morphology, chemical composition, phases, composite powder microstructure and porosity are mainly described.

In addition to the powder characteristics, the microstructure of the thermal sprayed coating was investigated and the dry abrasive wear resistance of coating was studied.

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2. EXPERIMENTAL

2.1. Characterization of composite powder

The WC-Co composite spray powder was produced using mechanically activated synthesis.

The granules size and their distribution in the studied powders were examined with the help of three analysing methods:

- Sieve analysis (–45 … +20 μm);
- Laser diffraction analysis by Laser particle sizer Analysette 22 Compact;
- Image analysis based on the Image-Pro Plus 3.0 system and the corresponding data processing programs.

For particle structure and composition analysis, cross-section polishes were made by mechanical grinding-polishing procedure. The best results were obtained by a fluid (not viscous) cold mounting by epoxy-based compound. The powder was mounted by help of Buehler vacuum impregnation system. Due to the high hardness of WC particles, diamond grinding-polishing was used.

The microstructure and morphology of the powder granules were investigated by the scanning electron microscope (SEM) Jeol JSM-840A using the secondary and backscattered electron imaging. Quantitative results of particles morphology were obtained by image analysis method based on the SEM cross-section photos.

The element composition of powder particles was studied by the energy dispersive X-ray microanalysis (EDS) on the Link Analytical AN10000 system. To evaluate element distribution inside powder particles, the X-ray mapping technique was used. According to the results, the resolution of element distribution is 0.5 – 1 μm.

Carbon content in the spray powder was measured by Elementar Analysensysteme GmbH firm element analyzing system Vario EL V2.6.

Chemical composition was alternatively tested by spark analyzer Spectrolab M. The spray powder was mixed

with iron powder and pressed. As a result, the composition of pressed compact material was heterogeneous and the obtained chemical composition measurements were inaccurate. Therefore, in further experiments the induction melting in the argon environment will be used.

Porosity measurements of the powder granules and coating were performed with the help of SEM, optical microscope and image analysing system.

The phase composition study of the investigated samples was carried out on Bruker D5005 X-ray diffractometer (XRD), using Cu K_a radiation at 40 kV and 40 mA. The range was 11 – 70° by step 0.040° and step time was 3 seconds.

2.2. Thermal spraying of coating

The HVOF spraying system TAFA JP 5000 (Praxair Tafa, USA) with fuel – kerosene was used for the deposition of coating. Spray parameters applied for the spraying of coating are shown in Table 1.

Table 1. Spray parameters for HVOF system

Parameters	TAFA JP 5000
Fuel and gases flow, l/min	Kerosene 0.38 Oxygen 1066
Fuel and gas pressure, kPa	Kerosene 828 Oxygen 938
Deposition rate, kg/h	9.0
Spray distance, mm	380

The coatings were deposited on the carbon steel plates (0.45 % C) as a substrate material. The plates should have the length, width and thickness of 60 mm, 35 mm and 10 mm, respectively. Coating thickness after spraying was ~ 300 μm.

2.3. Hardness and wear resistance study

The cross-sections of the sprayed coating were prepared to study the microstructure and Vickers micro hardness [4]. The different indentation loads were used.

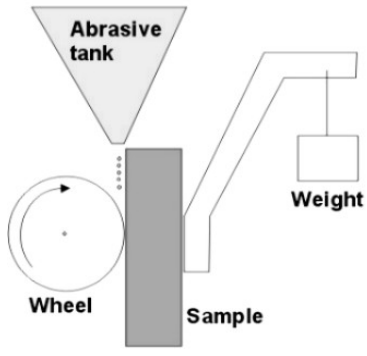


Fig. 1. Dry abrasive tester (ASTM G 65 – 94)

Dry abrasive wear resistance test was performed according to ASTM standard G65–94 (Fig. 1) [5]. Abrasive – quartz sand with particle size 0.1 – 0.3 mm was used. The amount of the sand was 3.0 kg. Disc diameter was 228.6 mm, the speed was 2.4 m/s, distance 1440 m and force 222 N.

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1. WC-Co based composite spray powder granules size and morphology

The classification of powder granules was performed by sieving. As the preferable size of granules is from 20 to 45 μm, the determined fraction was used to have high productivity of spraying and to avoid the oxidation processes. Due to the production method the powder granules are elongated in shape (Fig. 2).

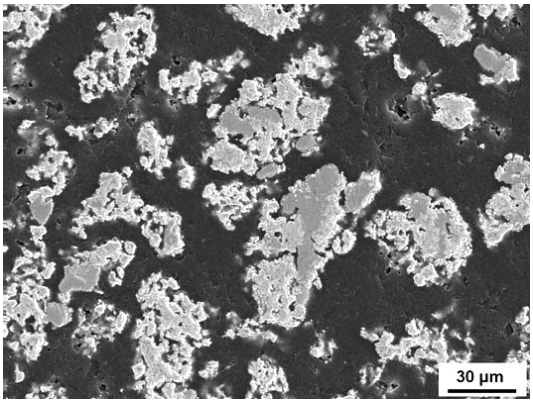


Fig. 2. SEM micrograph of the powder granules' cross-section

Therefore, the values for the granules' size measured by laser diffraction analysis was bigger. The arithmetic mean diameter was 43 μm; 28 % of the granules measured were over 50 μm and 5 % were less than 20 μm (by volume distribution). Granules larger than 45 μm are going through the sieve because of their elongated shape. Granules' size distribution is shown in Fig. 3 [6].

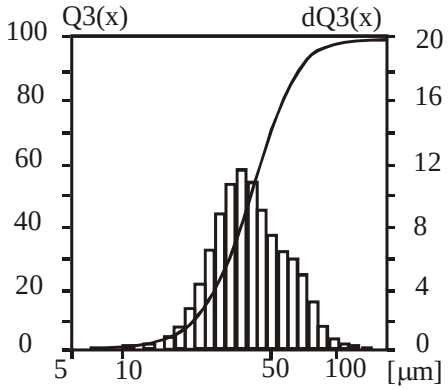


Fig. 3. Powder granules size distribution measured by laser particle analyzer

The granules should be spherical because they have to flow and elongation gave them a larger surface area, which influences to the conditions of melting and oxidation of powder particles.

3.2. Composition and microstructure of WC-Co based composite spray powder

Granules consist of many phases and were studied by XRD, SEM photos, X-ray mapping and by chemical composition.

By the XRD analysis, it was confirmed that granules consist of the following phases W, WC, Co_3W_3C and CoO. XRD analysis does not show the Co-phase because the XRD is not sensitive to cobalt (measuring parameters are given above, in Experimental) and/or it is amorphous (Fig. 4).

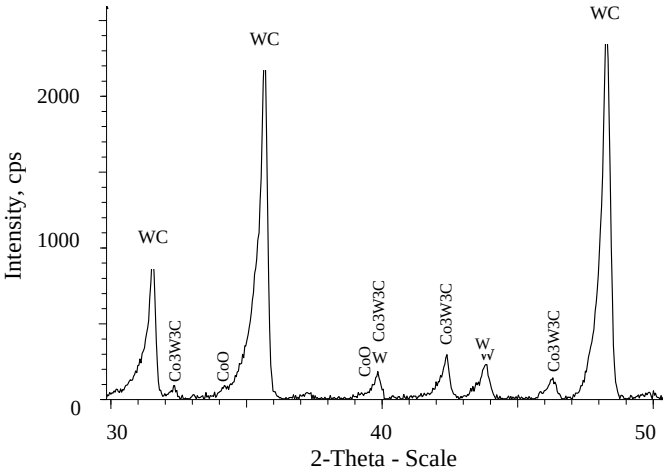


Fig. 4. XRD phase analysis of composite powder

Fig. 4 shows that the granules are mainly formed by help of desired sub-micrometric size WC particles. SEM photographs, X-ray mapping and hardness testing confirm that pure Co is one of the main phases. Co_3W_3C is formed because of the low carbon content on the powder (under 6.1 %) [7, 8].

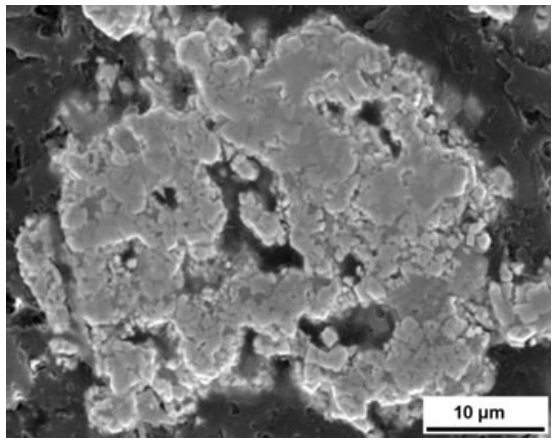
Micro- and sub-microstructure has an effect on the mechanical properties and wear resistance of the coating. Microstructure studies were made in the magnification of 300 times of the cross-section SEM photos (Fig. 2).

W and Co have different tones under SEM because of their atoms' different load. It was the basis for analysis with Image Pro 3.0 granules' porosity and phases. From the whole area, 12.1 % was porosity, 13.7 % Co and 74.3 % were WC sub-micrometrical particles in Co matrix. From the phase area, Co (8.9 g/cm^3) and WC-Co (14.5 g/cm^3) were calculated to mass percent where Co was 10 % and WC-Co 90 %. Initially, the material contained 15 % of Co, which means that 5 % of Co is in the WC-Co matrix and in the form of Co_3W_3C and in CoO.

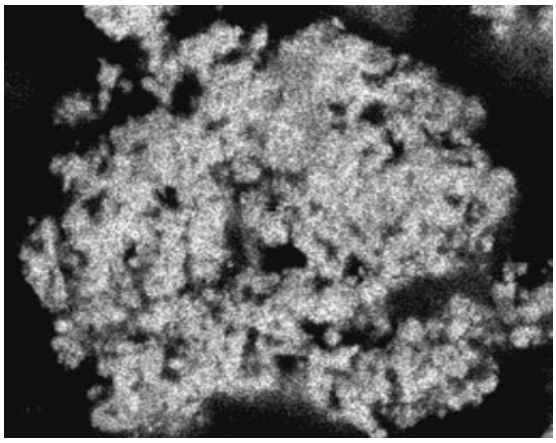
Sub-microstructure of the granules was observed, using magnifications of 8000 – 15 000 times. Fig. 5, a, is shown the sub-microstructure of powder granule. WC particles are mainly less than 1 μm in size and equally distributed in Co matrix.

The next step should be to analyse particle shape and size with Image Pro 3.0 to get a comparable numerical data for a next experiments to study the coating properties.

The mapping pictures in Fig. 5, b (W), Fig. 5, c (Co) and Fig. 5, a (SEM picture of same area) are above all to confirm the understanding of the SEM picture phase tones. But the mapping picture resolution and quality of image analysis is not as good as in SEM picture. Therefore, for the exact image analysis, SEM photos give more information.



a



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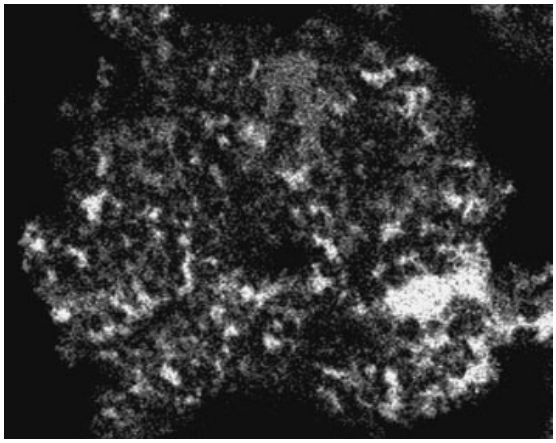

c

Fig. 5. Composite particle examined by mapping: a – SEM photo; b – W distribution; c – Co distribution

3.3. Characterization of coating microstructure

While the thermal spray powder granules consist of micro- and sub-microstructure, the coating structure can be observed from macrostructure up to sub-microstructure.

The microstructure is lenticular or lamellar, resulting from the rapid solidification of small globules, flattened from striking on to cold surface at high velocities [9].

As follows from Fig. 6 the coating macrostructure's contains a layer with large particles rich in Co (black areas on Fig. 6).

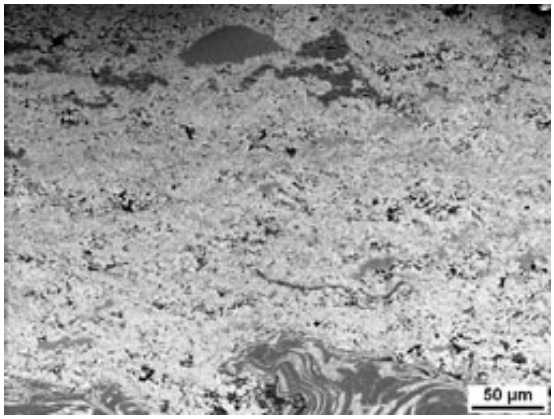

Fig. 6. SEM photo of the coating macrostructure (thickness of coating approximately 300 µm)

The layer with large Co particles most likely resulted from spraying thermal processes, because the upper layer cool down more slowly.

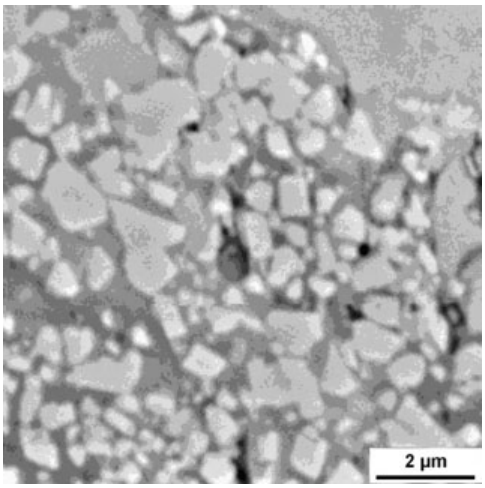

Fig. 7. SEM photo of the coating sub-microstructure

Sub-microstructure of the coating is similar to the powder granules – WC particles are not grown in coating process. The WC particles' size is mostly less than 1 µm (Fig. 7).

To compare the coating with other experimental coatings, a numerical size and shape analysis about sub-microstructure is needed because the WC particles size and shape have an important role in abrasive wear.

To analyze the coating's Vickers hardness, one- and two-dimensional distribution and different loads were used.

One-dimensional hardness measurements from cross-section distribution were made with 50 µm step and two different loads (0.98 N and 2.94 N). The results are given in Fig. 8.

The results are different: with 2.94 N of load, the hardness is sensitive to the softness of the zone rich in Co while with 0.98 N it is not.

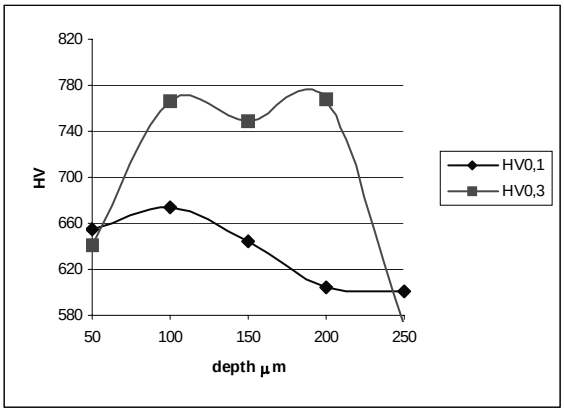

Fig. 8. Hardness distribution across the coating

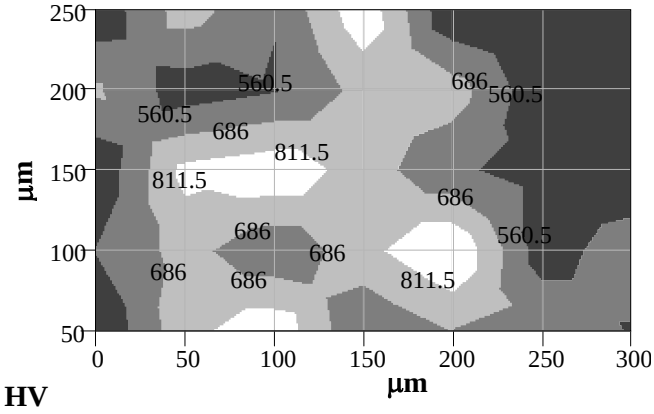

Fig. 9. Schematic illustration of the coating cross-section hardness measurements. The real microstructure is given in Fig. 10

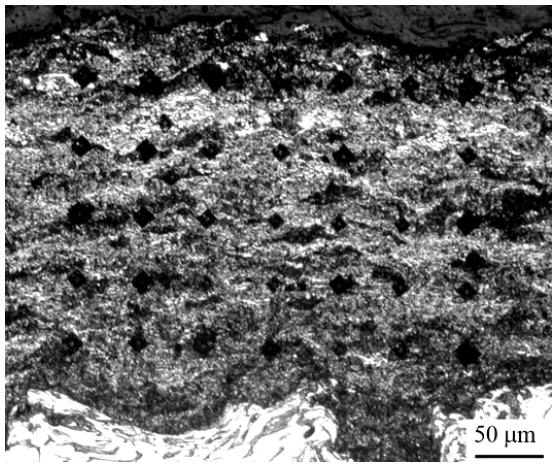

Fig. 10. Hardness measurement marks (see Fig. 9) and the coating microstructure made with light microscope

The two-dimensional hardness was measured with 0.98 N load and with 50 µm step. The results are given in Fig. 9 and the real structure of coating in Fig. 10. In Fig. 9, white areas indicate higher hardness and dark areas lower hardness. The two-dimensional hardness distribution may be interpreted as three-dimensional hardness distribution, which can give information about mechanical properties.

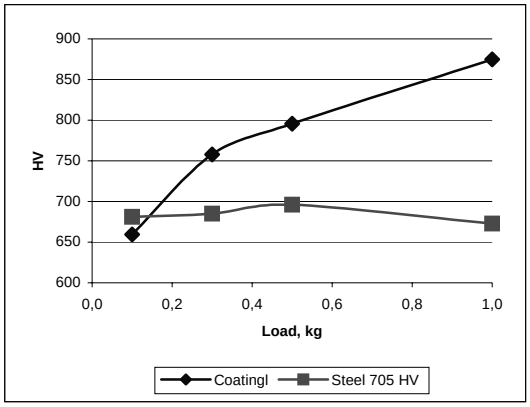

Fig. 11. Influence of the indentation load on Vickers hardness

As the first experiment with different loads gave different hardness values, hardness investigations with different loads (0.98 N, 2.94 N, 4.9 N and 9.8 N) were carried out. The accuracy of the measurements was controlled with the reference hardness (hardened steel 705 HV). The results are given in Fig. 11: an decrease in hardness with the decreasing of the load is caused mostly due to the porosity.

3.4. Abrasive wear resistance of coating

The results of the rubber wheel abrasion tests demonstrated that coatings sprayed from the experimental powder have more than two times higher wear resistance compared to the reference coating (similar with composition) TAFA 1343V sprayed at the same parameters. The better wear resistance can be explained by the homogeneous sub-microstructure of coating.

4. CONCLUSIONS

1. To characterize the microstructure and sub-microstructure of composite powder granules, numerical values are needed, so that they could be compared with the results of other experiments.
2. To analyze macro-, micro-, and sub-microstructures, it is possible to use effectively classical methods, for example analysing phase and elemental composition on the basis of SEM photos.
3. WC-based coating from experimental powder deposited by the HVOF spray technique demonstrated a better abrasion wear resistance compared to the reference coating sprayed from the analogous commercial powders due to the homogeneous sub-microstructure of coating.

Acknowledgments

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