4th International DAAAM Conference
"INDUSTRIAL ENGINEERING-INNOVATION AS COMPETITIVE EDGE FOR SME"
29-30th April 2004, Tallinn, Estonia

DOUBLE-CEMENTED RECYCLED HARDMETAL BASED POWDERS FOR THERMAL SPRAY

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Abstract

The coal of present paper is to get low cost the hardmetal powders from used material with comparable properties by commercial produced (Tafa Inc., Sulzer Metco Inc., etc.) powders. Thereby the composition and structure investigation of recycled hardmetal base powders produced by disintegrator milling was carried out. The recycled powders were used for thermal spray of hardmetal coatings by the detonation method.
Powders were mechanically plated with Co, using different plating techniques and thermal sintering. Powder particles chemical elements composition was studied by energy dispersive X-ray microanalysis (EDS) and elements distribution inside of powder particles was investigated on particles cross-sections by X-ray mapping technology.
Keywords: *disintegration, recycling, sintering, double-cemented hardmetal powder, particles composition*

1. INTRODUCTION

In terms of product lifetime for engineering materials and parts the surface is of prime concern. This involves both corrosion behaviour and mechanical properties such as material wear. Potential area of application of hardmetal powders from used hardmetal is thermal spray (flame spray and fusion, detonation and arc spray). Particles size and their morphology (shape parameters) determine the main technological properties of powders and the properties of powder materials, the final product. Technological properties of powders (bulk density, flowability, surface area etc.) as well as the potential areas of their application depend on their granularity and morphology. In powder use for thermal spray, their preferable particle form is spherical to have high flowability of powders and optimal conditions of particles melting and their spraying.

Earlier studies showed that formation of hardmetal coatings depend strongly on the size of used hardmetal particles sizes in detonation. The coarser fraction is suitable as reinforcing phase for spray and fusion self-fluxing alloy based coatings [Kulu and Halling, 1998]. For the detonation spray was used finer powders from 30-80 µm. From the point of efficiency of detonation spray and laying of coatings with lower porosity the finer (30-40 µm) is preferred [Kulu and Zimakov, 2000].

In addition to the particles size the experiments for determination the influence of hardmetal particles shape (angularity) to erosion rate of different metallic materials show the essentiality of this parameter in powders shape characterization [Mikli et al., 2002]. In the article [Zimakov et al., 2003] the influence of powder particle shape (spherical, angular) on the erosion rate and the wear mechanism of different metallic materials and the influence of the shape of reinforcement – powder particles in composite spray fusion coatings on the erosion wear resistance of coatings (Table 1).

Table 1. Properties of thermal sprayed composite coatings

Coating type	Shape of hard phase particles	Hardness HV0.2	
		Metal matrix	Hard phase
NiCrSiB+25 wt%VK15	angular round	755 660	1400
NiCrSiB+25 wt%VK15	angular round	685 560	1400
85wt%VK15 +15wt%Co	epiaxial agglomerate	1350	1350

Using of the commercial produced powders in thermal spray thick wear resistant detonation coatings (porosity less than 1 %) will be achieved. In the case of using recycled hardmetal powder in formation of detonation coatings many problems will be arise. Earlier studies show [Kulu et al., 1999], that hardmetal powder particles must be smaller than 100 µm. In the other case obtained coatings are very porous and uninformed. Additionally to the influence of particles size and shape to the formation of the coatings, the hardmetal particles must be covered with cobalt layer, which is a cement material between hardmetal particles in detonation coatings.
In earlier studies the improvement of the cohesion characteristics of powder particles in thermal spray was achieved by plating the hardmetal chemically by Co layer. The deposition of cobalt layer from $CoCl_2$ was used [Mikli et al., 2000]. As a result the obtained particles were built up with small WC-Co particles, but additionally to the Co they were joined by iron (Fig. 1). As a result the investigations were directed to improvement of Co deposition onto hardmetal particles (using electrolytic deposition, Co plating technique, etc.). Also the hardmetal particles were classified after disintegration by their sizes.

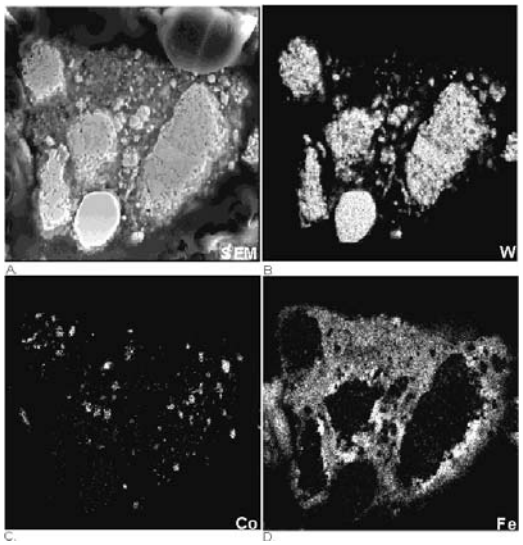

Fig. 1 **Powder D: A- SEM image; B- X-ray image of W; C- X-ray image of Co; D- from Fe** [Mikli et al., 2000]**.**

2. EXPERIMENTAL

Our analyses were carried out on WC-9Co hardmetal powder produced by milling with disintegrators DSL-158 and DSL-160.

2.1. Methods and powders used in double-cemented powders preparation

In the present study three different methods are used in preparation of double-cemented WC-Co powders. The experiments characteristics are given in tables 2-4. The tables showed the composition and size of initial particles; loaded pressure and annealing pressure of mixed powders.

Table 2. Mechanically mixed powders

Chemical composition	Size of particles, μm	Loaded pressure N/mm^2	Sintering temp., °C
1. 91 WC+9 Co	WC- 20-45 Co- 50	80	850
2. 17 WC+83 Co	WC- 20-45 Co- 50	80	850
3. 91 WC+ 9 Co	WC- 20-45 Co- 50	-	1000
4. 87 WC+ 13 Co	WC- 20-45 Co- 20	-	1150
5. 87 WC+ 13 Co	WC- 20-45 Co- 20	-	1300
6. 85 WC+ 15 Co	WC-4 (mean) Co- 2 (mean)	80	1300

Table 3. Powders, mixed in attritor mill

Chemical composition	Size of particles, μm	Loaded pressure N/mm^2	Sintering temp., °C
7. 87 WC+13 Co	WC- 45-90 Co- 20	-	1300
8. 87 WC+13 Co	WC- 20-45 Co- 50	-	-

Table 4. Powders, mixed in ball mill

Chemical composition	Size of particles, μm	Loaded pressure N/mm^2	Sintering temp., °C
9. 87 WC+13 Co	WC- 20-45 Co- 50	-	-
10. 87 WC+ 13 Co	WC- 20-45 Co- 4 (mean)	-	-
11. 85 WC+ 15 Co	WC- 20-45 Co- 20	-	1280
12. 85 WC+ 15 Co	WC- 20-45 Co- 20	-	1310
13. 85 WC+ 15 Co	WC- 20-45 Co- 20	-	1400

The end product- double-cemented powder was obtained after mechanical milling of above described powders. Attritor and ball milling were used to "glue" the Co particles on the surface of hardmetal particles. In the case of attritor milling the velocity of rotations 800 r/min for powder 7 and 168 r/min for powder 8 were used. The milling time was 1 hour. In the case of ball milling the velocity 81 r/min was used. The samples were studied after 2-72 hours of milling.

2.2. Methods used in double-cemented powders characterization

The powder particles and their cross-sections were studied in scanning electron microscope (SEM) Jeol JSM-840A.

The powder particles element composition was studied by EDS analysis. The LINK ANALYTICAL AN10000 system was used. To evaluate the elements distribution inside of powder particles X-ray mapping technique was used. It gives the resolution to element distribution approximately 1 μm and is enough in the case of 20-100 μm size particles.

3. RESULTS AND DISCUSSION

Using of mechanical activating (attritor and ball milling) in preparation of double-cemented hardmetal particles no significant mixing of WC and Co particles was observed. Only after high temperature treatment of milled powders the fusion of WC and Co particles was detected. At the sintering temperature 1280 °C (powder 11) the hardmetal powder was minimally covered with Co. Better results were obtained at 1310°C sintering temperature (powder 12). Obtained double-cemented powders structure was porous, but the initial WC and Co particles were quite heavily alloyed to each other. Sintering temperature 1400°C (powder 13) was too high. Obtained WC-Co substance was heavily fused and it was impossible to separate the joined particles.

Best results were obtained mixing mechanically the powders and using the compressing of mixed powders before thermal treatment. Similarly to the experiments carried out in ball milling the sintering temperature less than 1300 °C was not enough to achieve the wished-for results. End product from powders 1-4 was similar to the initial powders. No significant fusion of hardmetal and Co was observed. Sintering temperature 1300 °C (powders 5 and 6) caused the weak alloying of WC and Co particles, enough for further separation of substance into particles with desired size.

The compression of powders before thermal treatment was useful in reduction of porosity of double-cemented powders. Additionally to the compression and thermal treatment the size of initial particles influenced to the quality of end product. On the present study mainly the preliminary sieved 20-45 and 45-90 μm size fractions of hardmetal powder were used. The best results were obtained using the mix of micropowders (powder 6), where the average size of hardmetal particles was 4μm, Co

particles 2 µm, compression 80 N/mm^2 and sintering temperature 1300 °C. In figure 2 the structure and elemental composition of double-cemented powder 6 is presented. To obtain the comparable results with commercially produced Tafa powder the SEM micrograph of Tafa 1343 powder is presented in the Fig. 2, B. In comparison the powder 6 and Tafa powder (Fig. 2, A, B) the structures of that powders are similar. In the chemical composition Tafa powder consists only of WC and Co particles. Powder 6 consists additionally Ti (Fig. 2, F) and Fe (Fig. 2, E). Ti becomes into powder from the initial material (WC-Co and WC-Ti hardmetals were used). Fe becomes into structure from the hardmetal disintegration process due to interaction between hardmetal particles and disintegrator parts (made at steel). As a result of thermal sintering the Co and Fe are formed a separate phase (Fig. 2, C, E).

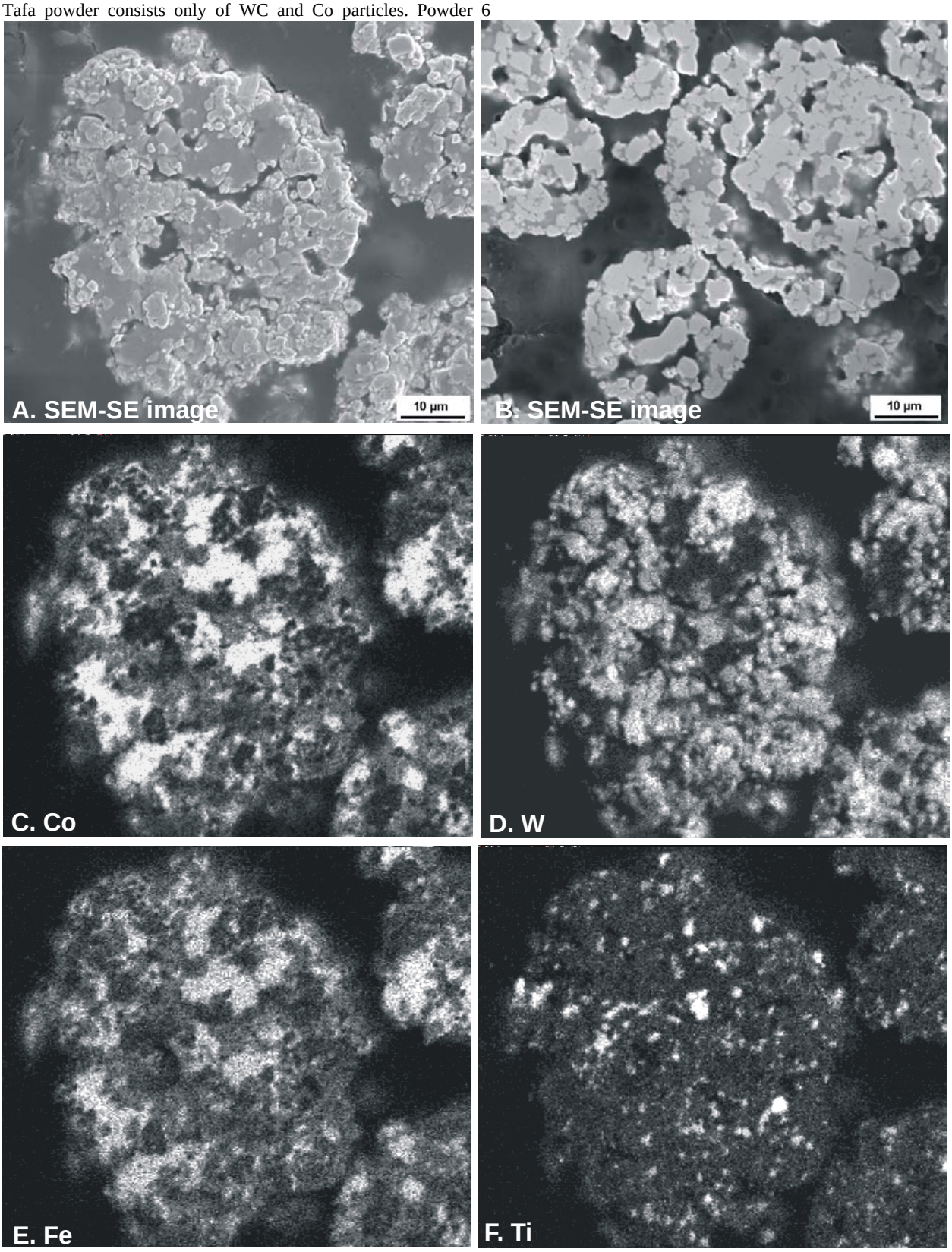

Fig. 2 SEM images and X-ray maps of studied powders: A- SEM image of powder 6; B- SEM image of commercial Tafa 1343 powder; X-ray images of powder 6: C- Co; D-W; E- Fe; F- Ti

4. CONCLUSIONS

1. To obtain the double-cemented hardmetal particles for thermal spray the different mechanical preparation techniques are applied.

2. Ball and attritor milling didn't give the significant influence in gluing process of Co particles on the top of hardmetal surface.

3. Using of mechanical mixing of near the micron size initial hardmetal and Co particles, further compression of mixture at 80 N/mm^2 and thermal sintering at 1300 °C the double-cemented powder, comparable with commercially produced is obtained.

Acknowledgement

The authors thank the Estonian Science Foundation for providing funding to perform work presented in this paper, grants No. 5891 and 5881.

5. REFERENCES

Kulu, P. and Halling J. (1998) *J. Thermal Spray Technology*, Vol.7 (2), pp. 173-178.

Kulu, P. and Zimakov, S (2000) *Surface and Coating Technology*, 130, pp. 46-51.

Kulu, P., Pihl, T., Zimakov, S., (1999*) Proc. of Int.Conf. Baltrib'99* . Kaunas, pp. 311-318.

V. Mikli, P. Kulu, H. Käerdi. **(**2002) *Proc. Lithuanian Acad. Sci. Materials Science*, Vol.8, 4, pp. 430-433.

Zimakov, S., Pihl, T., Kulu, P., Antonov, M, Mikli, V. 2003, *Proc. Estonian Acad. Sci. Eng*., 9/4, pp.304-316.

Mikli V., Kulu P., Käerdi H. (2000) *Proc. 2rd Int. DAAAM Conf*., Tallinn, pp. 201-204