

PRELIMINARY COMPARISON OF HARD METALS OBTAINED BY SPS AND BY ELECTRICAL RESISTANCE SINTERING (ERS)

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ABSTRACT

Cutting tools with improved properties and using a new processing method are being developed for both aerospace and automotive applications within EFFIPRO; an EC granted project. The aim of the project is to develop a new energy efficient sintering process for cutting tools production. This new method is based in Joule effect sintering process which allows extremely fast sintering cycles.

This work presents the preliminary results obtained during the characterisation of materials obtained using different sintering processes. A comparison of samples obtained by Spark Plasma Sintering (SPS) and Electrical resistance Sintering (ERS) is reported.

It was also found that the sintering conditions for ERS are strongly influenced by the electrical conductivity of the powders. Samples obtained by SPS and ERS show a microstructure and properties similar to commercial materials.

Keywords: Electrical sintering, ERS, FAST, cutting tools, tungsten carbide

1. INTRODUCTION

For over 70 years, WC–Co cemented carbides have been widely used for cutting tools of various materials and other machine parts which are required to show a high resistance to frictional wear. Their mechanical properties can be modified over a broad range by changing the content of the binding Co phase and the WC grain size [1].

Cemented carbides are usually produced by sintering with the participation of a liquid cobalt phase. However, the presence of this phase during the WC–Co sintering, stimulates the growth of the WC grains. [2]. Thus, the control of grain growth of the carbide phase during liquid phase sintering is an important objective. In general, decreasing WC particle size increases mechanical properties such as hardness, wear resistance, and transverse rupture strength of the composites [3]. Increasing the volume fraction of Co increases the fracture toughness at the expense of hardness and wear resistance [4, 5]. WC–cobalt and other similar cemented carbides are used as cutting tools because of a combination of desirable high hardness and high fracture toughness due to the respective contributions of the carbide and metallic phases.

The use of non-conventional sintering processes such as ERS (electrical resistance sintering) and SPS (spark plasma sintering) offer a unique opportunity to avoid the liquid phase sintering and thus limit the WC grain growth. During these fast sintering processes, electrical discharges remove

adsorbed gases and oxides from the powder particle surfaces, thereby facilitating the formation of active contacts between them. In effect, the process time is substantially shortened and the sintering temperature may be reduced.

This will allow the development of new or improved WC particulate composite cutting tool materials, offering an opportunity for innovative materials processing and property development along with potential commercial applications.

This work presents the preliminary results obtained within EFFIPRO, an EC granted project, using a novel sintering process (ERS: Electrical Resistance Sintering) and makes a comparison with conventional Sinter-HIP process and SPS process.

2. MATERIALS AND METHODS

WC-Co powder (submicron size) was used with the following composition: 6 wt% of Co. Powder was procured from a commercial source without any kind of organic binder (wax). The morphology of the powders can be observed in Figure 1. The shape of the particles is irregular and the particle size is less than 1 micron. The EDS analysis of the powder shows the clear presence of the WC and Co.

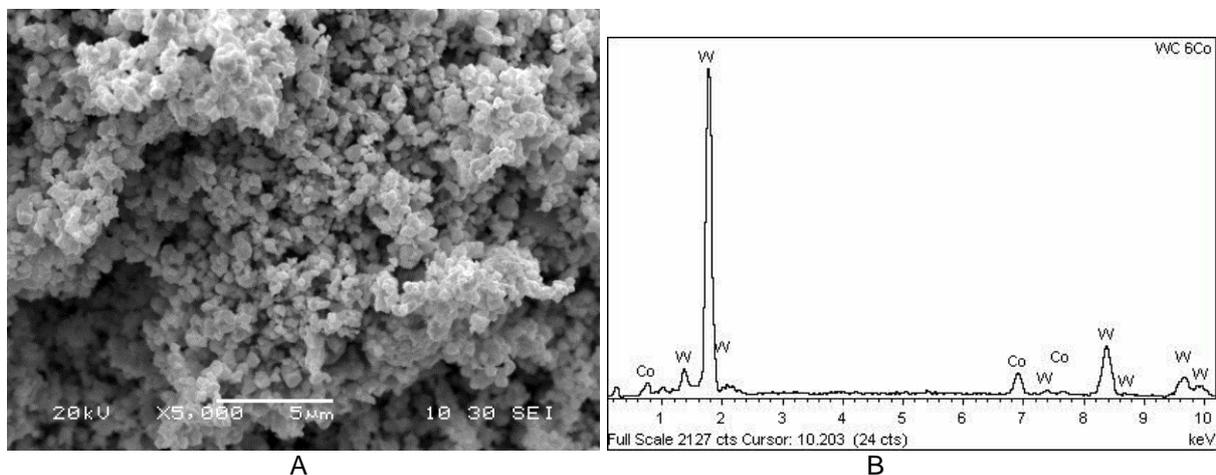


Figure 1. WC-6Co powder, A) SEM image, B) EDS analysis

For SPS processing, the powder was filled in a graphite die between two graphite punches. The compacts were fabricated by fast hot pressing using FCT-HP D 5/2 by FCT Systeme GmbH, Germany. The diameter of the samples was 30 mm. During the tests, the chamber was maintained under vacuum (10^{-2} Pa). Temperature was controlled by a pyrometer that measures the temperature in the interior part of the graphite punch. Maximum temperature was 1250°C with a holding time upto 2 min. The load applied was 40 MPa.

For the ERS processing, the powder was filled in a ceramic die between two electrodes (see figure 2). The diameter of the samples was 12 mm. The maximum applied current was around 9kA with a holding time of 690ms reaching a temperature around 1300°C. Maximum load was 100 MPa. The whole process duration is a few seconds.

Properties were compared with a conventional material produced by Sinter-HIP: After pre-treatment the powder was dry pressed and sintered in a Sinter-HIP furnace at a temperature of 1400°C under an argon pressure of 8 MPa (80 bar) for 90 min.

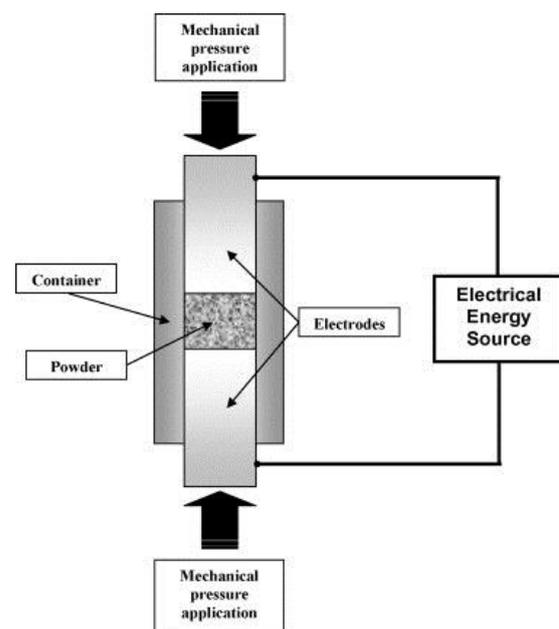


Figure 2. ERS set up drawing

Density (porosity) was measured by Archimedes method using a Mettler AE 240 weight balance. Metallographic determination of porosity and free carbon were performed according to ISO 4505. Microstructure and semi-quantitative chemical composition were analysed by optical and SEM microscopy (Jeol JSM 5910 LV microscope with an Oxford Inca 300 EDS accessory and Zeiss EVO50). Hardness Vickers (HV30) was measured according to the standard UNE-EN ISO 6507-1:2006. K_{IC} was calculated from the length of the radial cracks originating in the corners of the Vickers indentations according to the formula proposed by Shetty et al. [6]. Usually, five indentations were used to determine hardness and fracture toughness.

3. RESULTS AND DISCUSION

By means of the three processing routes (Sinter-HIP, SPS and ERS), samples were prepared for the analysis of density, microstructure, hardness and fracture toughness.

Density:

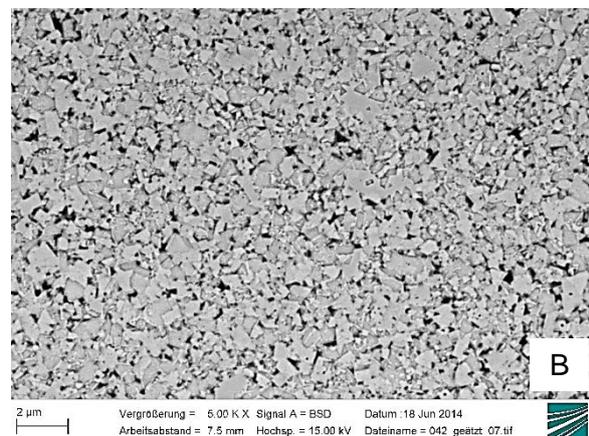
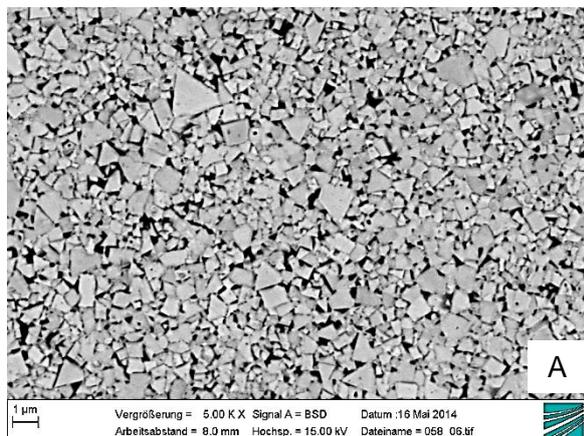
The density obtained in the three processing routes has been very similar. The best values are shown by the commercial reference. Nevertheless, samples obtained using the alternative sintering methods show very high density as well, within typical required values for hard metals tools.

Table 1. Density values

REF.	PROCESING	COMPOSITION	DENSITY (g/cm ³)	% of the Theoretical Density
1	Sinter HIP	WC-6Co	14,88	100
2	SPS	WC-6Co	14,83	99,7
3	ERS	WC-6Co	14,80	99,5

Microstructure:

Regarding microstructure, Figure 3 presents the microstructure of the commercial material (Sinter-HIP), the material obtained by SPS and the one obtained by ERS. In the three cases the microstructure is quite similar, polygonal WC grains can be appreciated surrounded by Co metallic matrix. Nevertheless, it seems to be a slight grain refining as the duration of the sintering process decreases, from the longer duration process to the shorter one: Sinter-hip (3A), SPS (3B) and ERS (3C). In addition, the rapidly sintered samples show some AB-porosity less than 02.



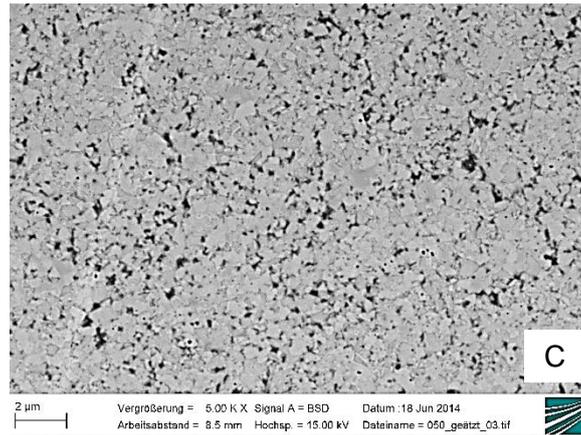


Figure 3. SEM images (etched with Murakami reagent) A) WC6Co sinter-HIP, B) WC6Co SPS and C) WC6Co ERS

Hardness & Fracture Toughness (K_{Ic}):

Table 2 shows the hardness and fracture toughness values.

Table 2. Mechanical properties

REF.	PROCESING	COMPOSITION	Hardness (HV30)	K_{Ic} MPa \sqrt{m}
1	Sinter HIP	WC-6Co	1833	9,6
2	SPS	WC-6Co	1938	9,4
3	ERS	WC-6Co	2080	10

Hardness Vickers (HV30) was analysed in the core of the samples. While SPS material presents slightly higher hardness than the Sinter-HIP reference, the hardness for ERS references showed an increase of 14% compared to the commercial reference.

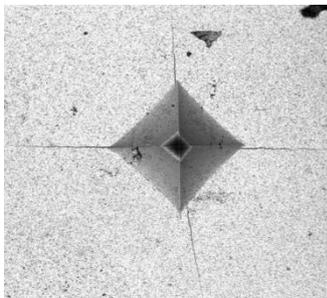


Figure 4. Example of the HV Hardness indentation and cracks propagation

Fracture toughness was determined using the PALMQVIST model:

$$K_{Ic} = 0.0889 * \sqrt{(HV * W)}$$

Where HV is the Vickers hardness and W is the Palmqvist crack resistance with the indentation load F and the mean radial crack length $4 * (c - a)$ according to

$$W = \frac{F}{4 * (c - a)}$$

Fracture toughness obtained using this method reveal similar behaviour for the three references, with values around 10 MPa m^{0.5}.

The very short processing time used in ERS (in the order of few seconds) helps in controlling the grain growth and thus obtaining finer microstructures. Although slightly lower densities are achieved with ERS process, it seems that the finer microstructure has an effect in improving the mechanical properties of WC-Co. The hardness as well as the fracture toughness gave better values than SPS or commercial references.

4. CONCLUSIONS

In this work the first results of the EFFIPRO project have been presented. The objective of this project is to develop a new very fast sintering method to produce cutting tools. Thanks to the short processing time, the novel sintering technology will enable, on the contrary to traditional furnace sintering methods based on thermal conduction and convection, an important energy consumption reduction and a fine control of material micro- and nanostructures.

Preliminary results regarding density, microstructure, hardness and fracture toughness were very encouraging: Using ERS it is possible to obtain materials with high density and more hardness and fracture toughness compared to the commercial (Sinter-HIP) and SPS materials. Taking into account that in all cases the same raw materials was used, it is believed that this increase is linked with the short processing time and the obtained microstructure. Microstructural investigations reveal the retained finer structure in ERS samples compared to the commercial and SPS samples.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

- [1] Kim BK, Ha GH, Lee DW. Sintering and microstructure of nanophase WC/Co hardmetals. *J Mater Process Technol* 1997;63:317–21.
- [2] Kim BK, Ha GH, Lee DW. Sintering and microstructure of nanophase WC/Co hardmetals. *J Mater Process Technol* 1997;63:317–21.
- [3] Zhang FL, Wang CY, Zhu M. Nanostructured WC/Co composite powder prepared by high energy ball milling. *Scripta Mater* 2003;49:1123–8.
- [4] Shin SG. Experimental and simulation studies on grain growth in TiC and WC-based cermets during liquid phase sintering. *Met Mater* 2000;6:195–201.
- [5] Ledoux MJ, Pham CH, Guille J, Dunlop H. Compared activities of platinum and high-surface-area Mo₂C and WC catalysts for reforming reactions. *J Catal* 1992;134:383–98.
- [6] Shetty DK, Wright IG, Mincer PN, Clauer AH. Indentation fracture of WC-Co cermets. *J Mater Sci* 1985;20:1873–82