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Fracture toughness of cemented carbides obtained by electrical resistance sintering

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INTRODUCTION

Cemented carbides (WC-Co) are materials used in a wide range of applications in many relevant industries, i.e. as cutting tools (turning, milling, drilling) for machining of metal components in the automotive and/or aerospace industry, as components of drill bits or road headers in the rock tools and mining area, or as wear parts in wire drawing dies or punch tools, all these applications with stringent requirements [1-3]. Regarding cemented carbides processing, the need to implement more efficient routes than conventional liquid sintering is one of the objectives pursued by the industrial sector. Field-assisted sintering techniques (FAST) have gained particular interest in the last decades [4-6] because of being very quick processes; particularly, the electric resistance sintering (ERS) process [7-9] consists in an electrical current passing through a powder mass to be sintered at the time that pressure is applied. The Joule effect acts heating and sintering the powders. However, despite its potential advantages, it remains an objective to control the variables associated with the sintering process, as well as to evaluate and rationalize the influence of these variables on the physical and mechanical properties of the manufactured samples.

The influence of the microstructural parameters on the behaviour in service has been widely studied by the scientific-technical community, particularly the mechanical and tribological performance [1, 10-16]. The content and physical dimensions of each constituent phase are the most common features for defining the microstructure [1,10,17]. Within this context, the principal parameters used to characterize the microstructure of hardmetals are the average grain size of WC particles (d_{WC}) and the binder volume content. However, both parameters are frequently varied simultaneously, and correlation between property and microstructure requires of additional two-phase normalizing parameters. Among them, the binder mean free path (λ_{Co}) is the most used one as it refers to the mean size of the metallic phase. In general, an increase of the binder mean free path implies a rise of the fracture toughness of the material at the expense of a decrease in hardness [17,18]. Main reason behind it is the fact that thicker and less constrained (i.e. effectively more ductile) ligaments exist for hardmetal grades with higher binder contents and coarser microstructures [14,15,17,19]. Also, the binder intercept size is an outstanding microstructural parameter because of its influence on the shear stresses of the material (for example, in cutting tool grades of hardmetals) [20].

Fracture toughness is the most important mechanical property of the WC-Co, considering the intrinsic fragility of these materials. There are different procedures to evaluate fracture toughness of cemented carbides [21-25]. The conventional indentation microfracture [24] is widely used in the literature because of its simplicity, cost and versatility. However, the measured values depend on the equation used, surface preparation, presence of residual stresses and the studied hardmetal grade [24,26,27]. In this context, the main objective of this work is to establish the relationship between the microstructure, the manufacturing process by ERS and the fracture toughness of WC-Co.

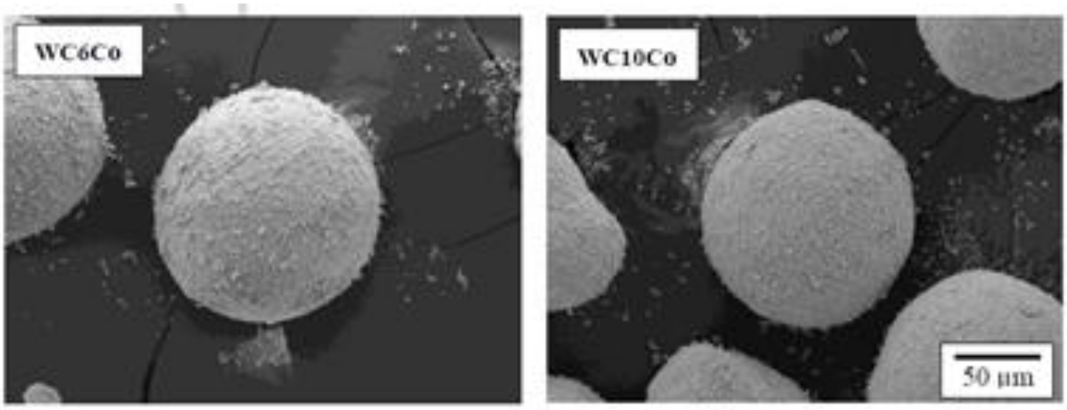


Fig. 1. SEM images of the powders used for the sintering of hardmetals.

Table 1. Chemical composition and properties of the starting powders supplied by Kyocera Unimerco (Denmark).

Grade	**WC-6Co**	**WC-10Co**
C (wt%)	5.78	5.52
O (wt%)	0.13	0.12
Spherical WC-Co particles (µm)		
$d_{10\%}$	86	78
$d_{50\%}$	141	128
$d_{90\%}$	225	204
$D_{[4,3]}$	148	136
Density (g/cm^3)		
Apparent	15.0	14.5
Tap	4.4	4.0
Flowability (s/50 g)	19.2	19.7
Compressibility (%) 100 MPa	61	63
Electrical resistivity (Ω·m) x 10^{-6}	6.9	6.4

Table 2. Experimental parameters associated to the electrical resistance sintering of the studied WC-Co.

Materials	
Cylindrical die	Alumina and sialon of high purity and density
Internal diameter	12 mm

Punches High purity copper
Wafers (in powder contact)
Cu-W alloy
ERS parameters
Compaction pressure 100 MPa applied at 100 mm/s
Continuous electrical
current wave
Pulse Square
Frequency (MHz) 10
Intensity (kA) 5 - 10
Time (ms) 300 - 1000

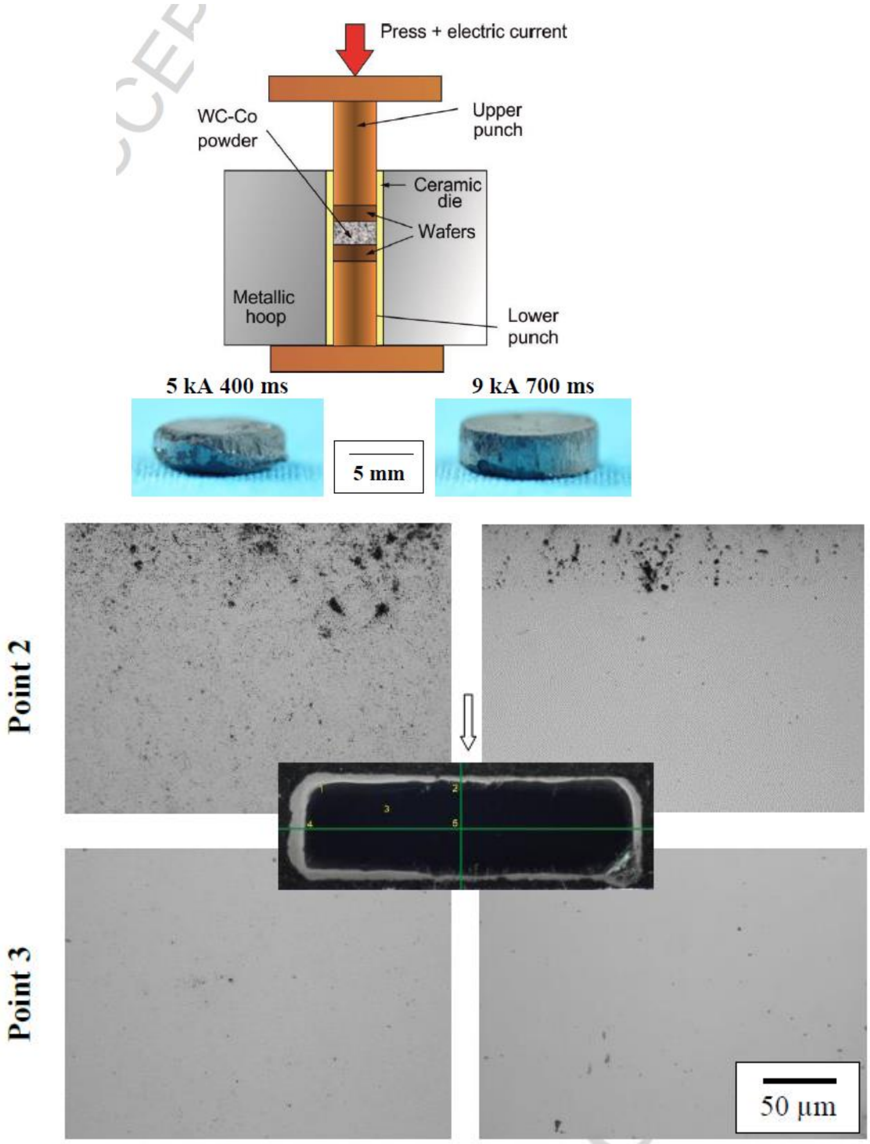


Fig. 2. Structural integrity (photographs) and porosity distribution (optical images – axial surfaces) of the pellets obtained in well apart sintering conditions with the alumina die.

Table 3. Experimental parameters of the microstructure of WC-Co studied.

Grade		**WC-6Co**	**WC-10Co**
f_{Co}	**wt%**	6	10
	vol%	10.2	16.5
WC-Co sintered	**Binder mean free paths (nm)** λ_{Co}	90 ± 8	120 ± 16
	Carbide contiguity ([33]) C_{WC}	0.64 ± 0.01	0.50 ± 0.02
	Carbide grain size (nm) d_{WC}	300 ± 30	290 ± 45

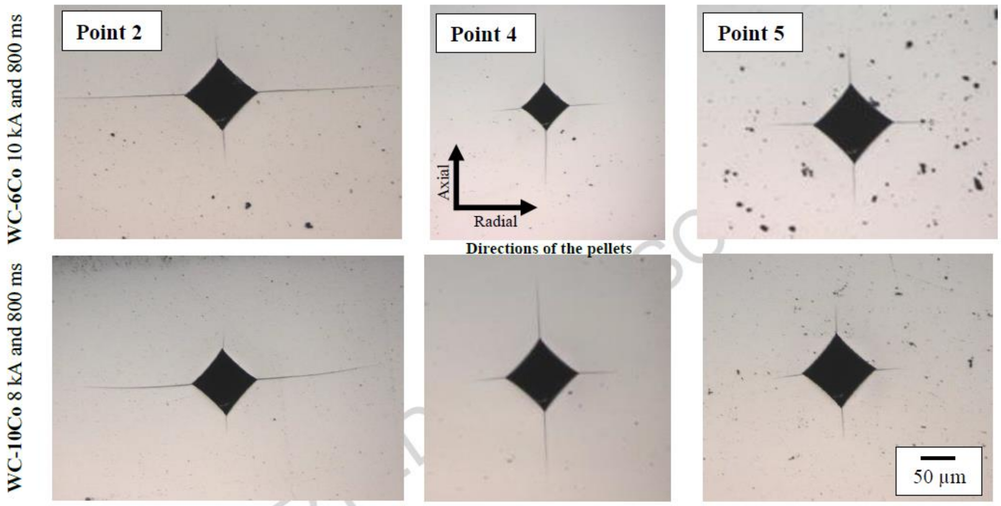
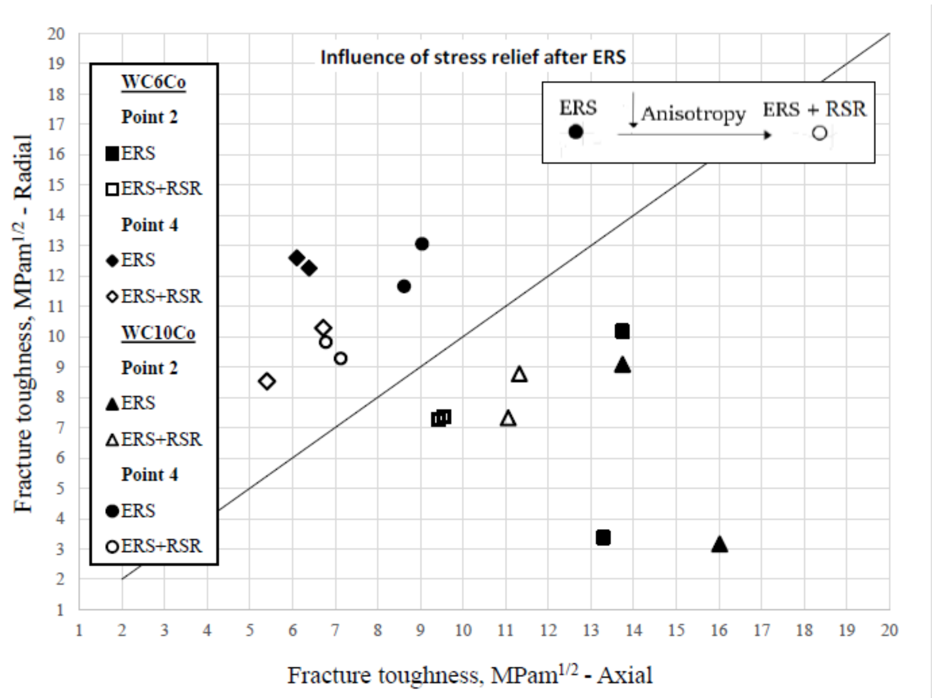


Fig. 5. Optical images of *Palmqvist* cracks induced from the corners of the *Vickers* indentations at different points (see scheme in Fig. 2) of an axial section of the WC-Co grades studied.



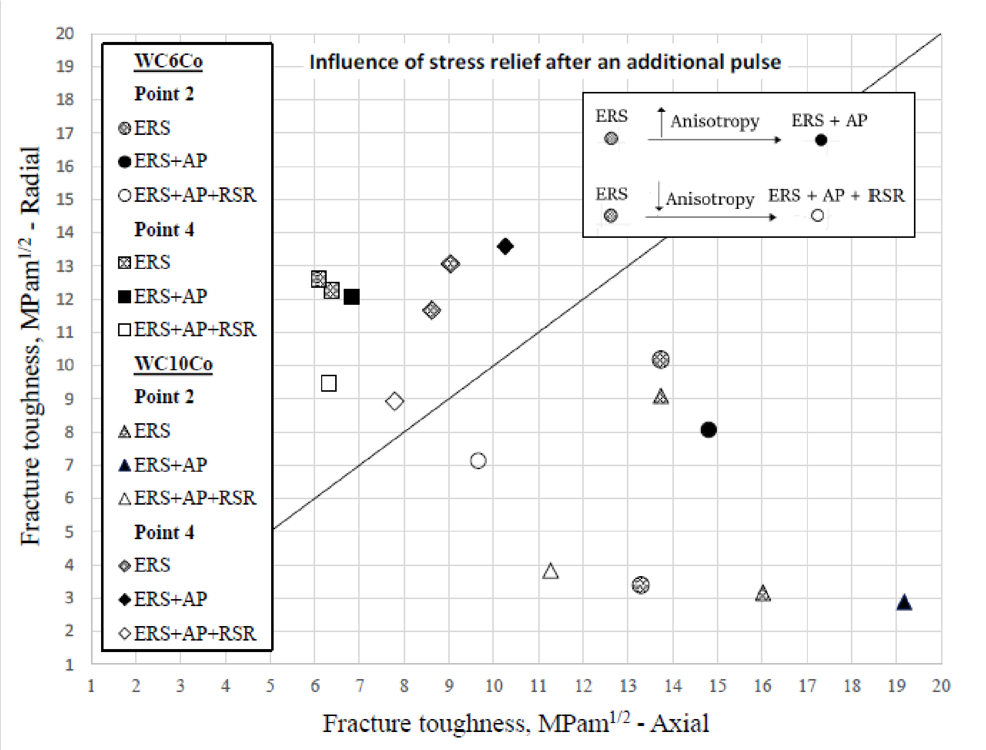


Fig. 10. Influence of AP and RSR treatments in the anisotropic mechanical behavior.

CONCLUSIONS

In this work two WC-Co grades obtained using the ERS technique were investigated. Based on the main findings of the study, the following conclusions may be drawn:

1) The ERS is a fast processing route. This technique is currently effective to obtain simple pieces or preforms of cemented carbides (WC-Co). The physical (density) and mechanical properties (hardness and fracture toughness) of the manufactured materials depend on the energy supplied during the electric sintering. This energy depends on the process parameters (sintering current and time, die materials, applied pressure, etc.).

2) The fracture toughness of these WC-Co depends on the role played by the cobalt ligaments and the deviation of the crack associated to the presence of the WC carbides (toughening mechanisms / R-curve behavior).

3) Electrically sintered WC-Co pellets present residual stresses, porosity and small microstructural changes (carbide grain size and cobalt binder thickness). These differences depend on the zone (bases and the lateral surface) and the direction (radial or axial), being the responsible of the anisotropy of the fracture toughness of the WC-Co pellets obtained by ERS.

4) The anisotropy of the mechanical behaviour is greater if additional electrical pulses are applied, while this heterogeneity is negligible if an adequate heat treatment of the WC-Co pellets is carried out following the ERS.

5) Whatever the WC-Co grade studied, in this work the following manufacturing process of the pellets is recommended: electric sintering (7 kA, 600 ms and 100 MPa, in an cylindrical alumina die), and then a relief treatment of residual stresses (800 ºC, 2 h and high vacuum).

This recommendation is made in terms of the best balance of structural integrity, density, homogeneity and mechanical behaviour (*Vickers* hardness and K_{Ic}) of the pellets.