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Development of electric resistance sintering process for the fabrication of hard metals: Processing, microstructure and mechanical properties

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ABSTRACT

This work presents the development of the Electrical Resistance Sintering (ERS) process for the fabrication of hard metals. The compositions of the materials produced were WC with 6 and 10 wt% of Co. In addition to the specific characteristics of the technology, the characterization of the produced parts is presented and compared to materials obtained by conventional processes.

The parts produced by ERS present densities comparable to the ones obtained by conventional methods. The microstructural comparison shows a considerable grain size reduction in the ERS materials which consequently brings a hardness increase. ERS materials show similar fracture toughness to conventional ones.

The very fast sintering allows performing the process without any protective atmosphere, therefore making this process very attractive for the production of materials that need to be sintered under non-oxidising environments. The total duration of the cycle, including heating, holding time and cooling is few seconds.

Finally, some considerations about the scale up and possible industrialization of the technology are explained.

1. Introduction

ECAS (electric current assisted sintering) gathers a family of consolidation methods in which mechanical pressure is combined with electric and thermal fields to enhance particle bonding and densification. The primary purpose of imposed electric current is to provide the required amount of resistive heat. The expression field-assisted sintering techniques (FAST) have become also popular in literature to denominate these technologies. Indeed, the speed of the processes is the most remarkable characteristic and the common factor among these techniques.

ECAS techniques can be classified with respect to the processing time [1]. In conventional ECAS processes, like SPS (Spark Plasma Sintering) [2], low current densities and voltage are applied (typically <10 V and 1 kA/cm^2). With these conditions, processing time is in the range of minutes and for that reason a controlled atmosphere is needed. However, in very fast processes like EDS (Electro Discharge Sintering) [3] and ERS (Electric Resistance Sintering) [4–6] higher current densities are applied (typically >5 kA/cm^2) and the processing time is few seconds. The short duration of the cycle permits the processing in air without any protective atmosphere. This is a very important advantage from the economic point of view. In EDS, the electric discharge is produced with a high-voltage capacitor bank, whereas in ERS the current is produced by a low-voltage transformer (around 10 V). Therefore, the current applied to the sample is easier to control in ERS and this improves the homogeneity of the products.

Electrical resistance sintering (ERS), was already described in 1933 by Taylor [7] and later modified by Cremer [8] in 1944. However, up to now, the ERS process has been used for the consolidation of high conductive materials (pure metals) at lab scale, obtaining very small pieces [4–6]. The development of medium voltage machines (40 V) opens the possibility of obtaining larger parts and using materials with lower electrical conductivity like composites and cermets.

WC–Co cemented carbides have been widely used for cutting tools of various materials and other machine parts which are required to show 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 [9–13]. 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. In general, with decreasing WC

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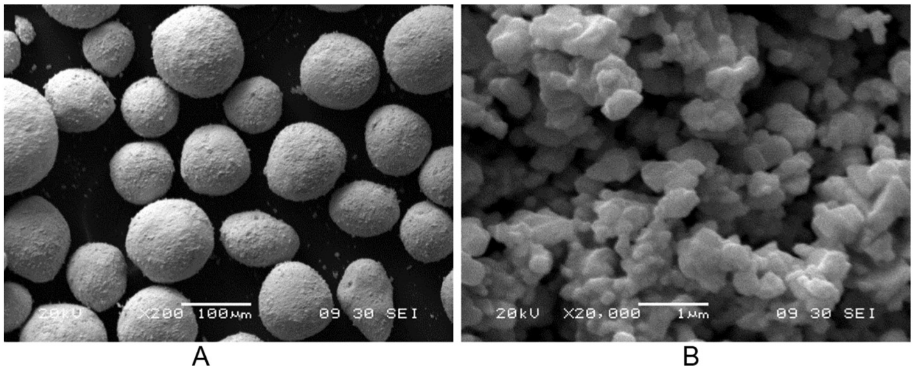

Fig. 1. WC-6Co powder, A) SEM image of the granulated powder, magnification ×200, B) SEM image of the surface of the granules, magnification ×20000.

particle size properties such as hardness, wear resistance, and transverse rupture strength of the composite are improved [14–16]. The effect of the nanostructure in the WC-Co carbides properties has been extensively studied [17–19]. It is known that fracture toughness decreases with increasing hardness in conventional composites, whereas the increase of hardness in nano-structured composites does not further reduce their bulk fracture toughness. This implies that different dominant toughening mechanisms exist in the conventional and nanostructured composites. Very fast sintering processes offer a unique opportunity to avoid the liquid phase sintering and thus limit the grain growth.

This paper presents the development of the ERS technology for the processing of hard metals. The new pilot plant scale equipment is shown. An electric press of 15 tones was combined with medium voltage transformers in a fully automated ERS equipment.

Microstructural and mechanical characterization of the materials are presented and compared to commercial sinter-hipped carbides. In addition, some important aspects of the technology are described.

2. Materials and methods

WC-Co powders (submicron size) were used with the following composition: 6 wt% of Co and 10 wt% of Co. Granulated powders from a commercial source were procured with organic wax and a pre-treatment was performed in order to eliminate organic components. The spherical morphology of the granules with a size between 100 and 200 µm can be observed in Fig. 1A. Each granule is composed of very fine powder (size <1 µm). The surface of one granule is observed in Fig. 1B. Apparent density of the granulated powder was 3,7 g/cm^3.

For the ERS processing, the powder was filled in an alumina based ceramic die between two copper electrodes (see the diagram of the ERS machine in Fig. 2A). The dimensions of the produced parts were 22 mm in diameter with a thickness of 10–14 mm. The maximum applied current density was between 4 and 5 kA/cm^2 with a holding time of 500 ms. Maximum load was 100 MPa. An image of the prototype machine used in this work is presented in Fig. 2B. This fully automated ERS machine was developed within the EU funded EFFIPRO project. Maximum voltage of the machine is 40 V with a maximum current of 35 kA. The pressure is applied using a 15 tones electrical press.

Density was measured by Archimedes method using a Mettler AE 240 weight balance and porosity was analysed according to the standard UNE-EN ISO 4605:1978. Microstructure and semi-quantitative chemical composition were analysed by optical and SEM microscopy (Zeiss Nvision 40 and Zeiss EVO50). Hardness Vickers (HV30) was measured according to the standard UNE-EN ISO 6507-1:2006. KIC was calculated from the length of the radial cracks originated in the corners of the Vickers indentations according to the formula proposed by Shetty et al. [20]. The analysis of the magnetic coercivity HC and saturation

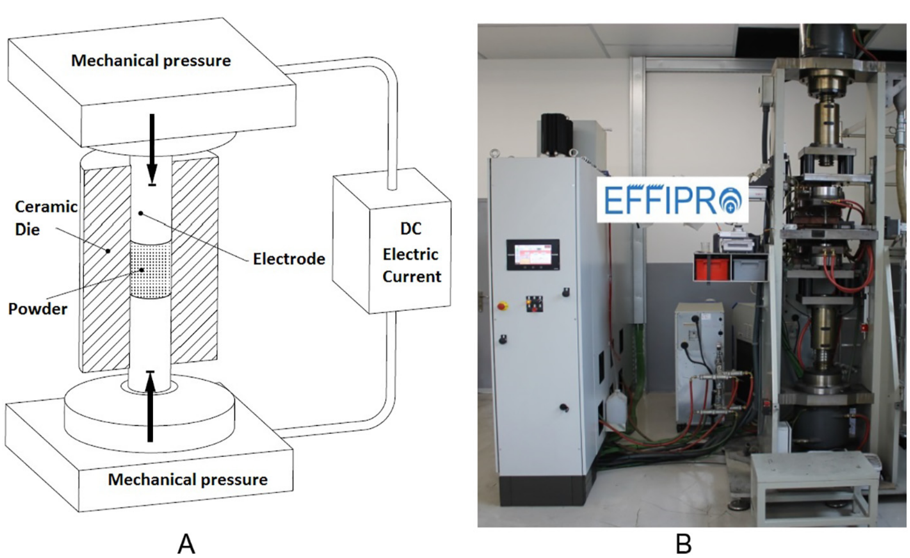

Fig. 2. A) Diagram of the technology, B) Prototype machine, developed within the EFFIPRO project.