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Implementation of an effective time-saving two-stage methodology for microstructural characterization of cemented carbides

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ABSTRACT

Linear intercept on scanning electron microscopy micrographs is the most commonly used measurement method to determine carbide grain size and contiguity in WC–Co cemented carbides (hardmetals). However, it involves manual time-consuming measurements and is critically dependent on the quality of the micrographs as well as on the identification and definition of grain boundaries. In this study a two-stage methodology for microstructural characterization of hardmetals is presented. First, a digital semi-automatic image analysis procedure for grain size determination of the carbide phase is presented. It involves an experimental assessment of grain size on processed images corresponding to a series of WC–Co and WC–Ni cemented carbide grades with different microstructural characteristics. Obtained results are then compared to the values obtained by means of the linear intercept technique. A good correlation between the mean grain sizes determined following both measurement techniques was attained. Based on experimental findings, a series of empirical relations were found to correlate grain size distributions obtained following both methods. Second, an empirical relation for estimating carbide contiguity in WC–Co cemented carbides is proposed. This relation considers simultaneously the influence of the binder content and the experimentally determined mean grain size on contiguity. The proposed equation for contiguity estimation is based on extensive data collection from open literature. An excellent agreement was attained between contiguity values estimated from such equation and those obtained using the linear intercept technique. This validates the two-stage procedure as an effective time-saving methodology for microstructural characterization of WC–Co cemented carbides.

1. Introduction

Mechanical and tribological performance of WC–Co cemented carbides, also referred to as hardmetals, is closely related to its particular microstructure, being the content and physical dimensions of each constituent phase the most common features for defining it (e.g. Refs. [1,2]). In 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 fraction (V_{binder}). However, both parameters are frequently varied simultaneously, and correlation between property and microstructure requires of additional two-phase normalizing parameters. Among them, the contiguity of the carbide phase, C_{WC}, and the binder mean free path, λ_{Co}, clearly stand out (e.g. Refs. [1, 3–5]). The former describes the interface area fraction of WC carbides that is shared by them [6], whereas the latter refers to the mean size of the metallic phase. In addition to these key parameters, there are other microstructural aspects that strongly influence the properties of cemented carbides, such as the amount of W and C dissolved in the binder [7] as well as the shape [8] and grain size distribution of the carbides [9]. Although the influence of such variables on the performance of hardmetals is widely recognized, normally they are not taken into account when analysing mechanical property–microstructure relationships [1].

The linear intercept (LI) method, also known as the Heyn method, is the most widely used protocol to determine grain size in WC–Co cemented carbides [10]. This method consists in drawing a series of horizontal parallel lines onto a scanning electron microscopy images, usually around 5 lines per micrograph, and then measuring the length of the carbides crossed by the lines. With this method, in order to reduce the mean grain size uncertainty below 10%, at least 200 intercepted grains have to be measured [10], and this number is even higher when measuring grain size distributions. Taking this into consideration, two main drawbacks are directly identified: excessive time-consuming

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associated with manual implementation, and uncertainty related to both image quality and identification/definition of grain boundaries.

In addition to LI method, other techniques have been developed to determine the grain size of hardmetals. Image analysis (IA) and electron backscatter diffraction (EBSD) methods are two successful examples. The former is based on the measurement of the grain areas in IA-treated micrographs [11–13], which are then converted to equivalent geometrical shapes. In general, determined surfaces are considered as circles and an equivalent circle diameter (ECD) is reported. ECD and LI methods describe similar trends; however, they result in different values. These differences are governed according to the relation (ECD) = 1.15(LI), allowing to convert the values in each case [14]. The EBSD approach consists in measuring the area of the carbides on the basis of relative differences on crystallographic orientation from grain to grain. With this technique very accurate results are obtained. Moreover, it is really useful when measuring the grain size of ultrafine and nano-sized grades. Although EBSD is an automated technique, it is slow and requires a really good surface preparation as well as a careful data treatment and analysis [15,16].

Similar to grain size, contiguity can also be estimated using the linear intercept method according to the following expression [1,3,4,17]:

$$C_{WC} = \frac{2N_{WC/WC}}{2N_{WC/WC} + N_{WC/Binder}} \quad (1)$$

where $N_{WC/WC}$ and $N_{WC/Binder}$ are the number of carbide/carbide and carbide/binder intercepted interfaces. Roebuck and Bennett [17] studied the contiguity as a function of the binder content (V_{binder}) for a series of hardmetals and proposed the following empirical relation:

$$C_{WC}(V_{binder})^n = D \quad (2)$$

where the best-fitting is obtained when n and D constants take values of 0.45 and 0.2, respectively. However, they pointed out a significant dispersion of C_{WC} values for each specific binder content, associated with different mean sizes and distributions of the carbide phase and to the difficulty to properly interpret all interfaces [1,9,17]. Furthermore, contiguity depends on additional factors such as the carbide particle shape [8,18] or the manufacturing conditions, including sintering time and temperature [19]. On the other hand, other authors sustain that contiguity is exclusively dependent on binder content, and consequently is not influenced by grain size [20]. This statement was based on an experimental investigation of contiguity, by means of the LI method, carried out in a wide range of hardmetal grades.

Following the above ideas, the objective of this work is two-fold. First, it aims to present a simple automated image analysis procedure for grain size determination in WC–Co and WC–Ni cemented carbides. The second objective is to propose an empirical relation for contiguity estimation as a function of mean grain size and binder content. The methodology is implemented in a series of hardmetal grades and results are compared to those measured by the linear intercept technique. The good correlation among them validates the feasibility of this two-stage methodology as an effective time-saving process for microstructural characterization of cemented carbides.

2. Materials and experimental aspects

2.1. Materials, sample preparation and measurements done by means of the linear intercept method

A set of seven hardmetal grades with binder contents ranging from 6% to 15% in weight and ultrafine, fine, medium and coarse carbide grain sizes was characterized (Table 1). All studied materials were experimental hardmetal grades supplied by Sandvik Hyperion. In doing so, samples were mounted in Bakelite, then ground and diamond polished up to mirror-like surface finish following a 6, 3 and 1 μm sequence, with a final colloidal silica stage. The same polishing time and applied load was used for all the studied materials. Subsequently, Field Emission Scanning Electron Microscopy (FESEM) micrographs were acquired (e.g. Fig. 1a) using a Jeol JSM-7001F unit. The magnification of the micrographs was selected so that they contained around twenty carbides in the horizontal direction. In order to reduce grain size uncertainty, at least 400 grains were measured for each investigated hardmetal grade. Additionally, contiguity (C_{WC}) was assessed following the linear interception method on the same micrographs according to Eq. (1).

Table 1
Nomenclature, binder chemical nature, binder volume content, binder weight content and grain size of investigated WC–Co and WC–Ni cemented carbide grades.

Nomenclature	Binder chemical nature	V_{binder} (vol.%)	V^{wt}_{binder} (wt.%)	Grain size
6UF	Cobalt	13.0	6.2	Ultrafine
10UF	Cobalt	17.5	10.5	Ultrafine
15UF	Cobalt	24.4	14.7	Ultrafine
9F	Nickel	16.5	9.0	Fine
11M	Cobalt	19.1	11.0	Medium
15M	Cobalt	25.0	15.0	Medium
10C	Cobalt	16.9	10.0	Coarse

2.2. Automated image analysis procedure

The first main objective of this investigation is to present a procedure for grain size determination from computer processed images. Within this context, FESEM micrographs were treated using the Fiji free available software by means of a series of algorithm operations: smooth, binarization, Euclidian Distance Map, and Find Maxima algorithm; as well as by the application of binary operations between processed images. The procedure followed to obtain binary images suitable to accomplish grain size automatic analysis is detailed below.

First, a smooth filter that blurs the original micrograph (Fig. 1a) was applied. It replaces each pixel with the average of its 3 × 3 pixel neighbourhood, preventing then small imperfections or dark spots within the phases when binarizing the image. Second, the grey-scale image was transformed to a black and white binary image (Fig. 1b). This was done by applying the GraphCut operation, which is based on the implementation of the max-flow algorithm, as reported by Boykov and Kolmogorov [21]. Here, a local grey-scale threshold is determined and the pixels darker than it are displayed as white, whereas the lighter pixels are displayed as black. The smoothness of the segmentation can be adjusted according to the application. Subsequently, a binary open–close operation was implemented to remove the "island pixels" surrounded by their opposites. Then, a Euclidian Distance Map (EDM) operation was performed (Fig. 1c). The function of the EDM algorithm is to label each foreground pixel with a grey value equivalent to its distance to the nearest background pixel. Thus, a grey scale image was obtained where the brightest points correspond to the carbide pixels furthest from the metallic phase. Following the EDM operation a Find Maxima algorithm was applied in order to segment the EDM grey-scale image into particles (Fig. 1d). The particle size is determined according to the intensity of the local maxima pixels. As a result, a binary image with black background and white lines at the grain boundaries was obtained. The final step consisted on a binary sum operation between the binarized image (Fig. 1b) and the micrograph resulting from the Find Maxima operation (Fig. 1d). Obtained micrograph (Fig. 1e) exhibited a strong similarity to the original micrograph. However, some grain boundaries were not recognized, i.e. there were a few carbide agglomerates that appear as a unique carbide grain, but in reality consisted of two or more carbide grains. In order to minimize this discrepancy, the treated image was overlapped with the original micrograph, and the missing grain boundaries were introduced manually. The final treated micrograph is shown in Fig. 1f.