Implementation of an effective time-saving two-stage methodology for microstructural characterization of WC-Co 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 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 cemented carbides grades with different microstrucural 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 are 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 binder content and 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 is 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.

KEYWORDS: WC-Co cemented carbides, carbide grain size, carbide contiguity, microstructure, image analysis

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 later 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 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 be also estimated using the linear intercept method according to the following expression [1,3,4,17]:

(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:

(2)

where the best-fitting is obtained when n and D constants take values of 0.45 and 0.2, respectively. However, they point 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 is 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 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 WC-Co 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 WC-Co cemented carbide grades with binder content ranging from 6% to 15% in weight and ultrafine, fine, medium and coarse carbide grain sizes was characterized (**Table 1**). 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. Subsequently, Field Emission Scanning Electron Microscopy (FESEM) micrographs were acquired (e.g. **Figure 1a**) using a Jeol JSM-7001F unit. 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).

Nomenclature	Binder content ($\%_{wt.}$)	Grain size
6UF	6	Ultrafine
10UF	10	Ultrafine
15UF	15	Ultrafine
9F	9	Fine
11M	11	Medium
15M	15	Medium
10C	10	Coarse

Table 1. Nomenclature, binder content and grain size of investigated WC-Co cemented carbides grades.

2.2. Automated image analysis procedure

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 (EDM), 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 (**Figure 1a**) was applied. It replaces each pixel with the average of its 3x3 pixels 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 (**Figure 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 is performed to remove the "island pixels" surrounded by their opposites. Then, a Euclidian Distance Map operation was performed (**Figure 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 (**Figure 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 (**Figure 1b**) and the micrograph resulting from the Find Maxima operation (**Figure 1d**). Obtained micrograph (**Figure 1e**) exhibited a strong similarity to the original micrograph. However, some grain boundaries are not recognized, i.e. there are a few carbide agglomerates that appear as a unique carbide grain, but in reality consist 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 **Figure 1f**.

Figure 1. Schematic representation of the image analysis procedure used to assess the carbide grain size of investigated materials. Presented micrographs correspond to: (a) initial FESEM micrograph, (b) binarization of the original image followed by (c) EDM algorithm, (d) Find Maxima operation, and (e) OR operation between the binarized original image (Figure 1b) and the micrograph resulting from the Find Maxima operation (Figure 1d). The final processed and manually corrected image is given in (f).

OR??

3. Results and discussion

3.1. Carbide grain size distribution

Grain size of carbides was estimated in processed micrographs by measuring the areas occupied by single particles and converting them in equivalent square side lengths??. This was done by considering that all the carbides had the same geometrical shape. IA measurements were performed in micrographs corresponding to both: final treated and corrected micrographs (**Figure 1f**), designed as IA – A, and final processed micrographs but without corrections (**Figure 1e**), named as IA – B. For both methodologies measurements corresponding to the carbides positioned at the edge of the images were discarded. A particular square shape was selected due to the proximity between the circularity of a square (circularity = 0.79) and the mean circularity measured for the carbides in the processed and corrected images (IA – A) (see **Table 2**). Circularity is a geometrical factor that determines how circular the shape of an object is. It is measured according to the following equation:

(3)

where A and P are the area and the perimeter of the object, respectively. Therefore, the circularity of a circle is equal to 1, whereas that of a line with infinite length is 0. In addition to the average size and distribution of the carbide phase, the shape of WC particles is also an important microstructural factor to characterize. In this regard, Shatov and coworkers have suggested that it may also have a significant influence on the mechanical properties of hardmetals [22]. Determined circularity for studied materials is shown in **Table 2** together with mean grain size and standard deviation values measured using LI and IA techniques.

Nomenclature	LI d_{WC} (µm)	IA – A d_{WC} (µm)	IA – B d_{WC} (µm)	IA – A Circularity
6UF	0.4 ± 0.21	0.39 ± 0.22	0.56 ± 0.25	0.69 ± 0.07
10UF	0.39 ± 0.19	0.42 ± 0.20	0.49 ± 0.21	0.67 ± 0.11
15UF	0.47 ± 0.22	0.46 ± 0.24	0.55 ± 0.27	0.68 ± 0.10
9F	0.77 ± 0.78	0.78 ± 0.47	0.97 ± 0.62	0.70 ± 0.11
11M	1.12 ± 0.71	1.29 ± 0.58	1.55 ± 0.76	0.70 ± 0.12
15M	1.15 ± 0.92	1.08 ± 0.59	1.29 ± 0.71	0.71 ± 0.08
10C	2.45 ± 1.37	2.39 ± 1.15	2.67 ± 1.40	0.72 ± 0.11

Table 2. Average grain size determined for investigated cemented carbides grades following the linear intercept and image analysis methods. Information on the circularity values obtained with the IA – A process is also included.

From **Table 2** it is interesting to point out that LI and IA – A measurement methods yield similar mean grain size values for studied materials, although there is not a clear correlation between them. On the other hand, and as expected, average grain sizes measured following the IA – B protocol tend to be slightly superior to those determined with the LI method.

An analysis of grain size distributions attained for the studied materials following the different investigated measurement methods was attempted. Relative and cumulative grain size distributions were plotted for the three investigated measuring methods and each studied hardmetal grade. In all the cases the number of selected bins for the distributions was 20, corresponding to the square root of the minimum number of measured grains. Relative distributions were correlated using a Gauss curve. Meanwhile, cumulative distributions were fitted using a Boltzman equation. In the first case, R^2 parameters of the order of 0.85 were obtained. On the other hand, quite high R^2 values (close to 0.99) were obtained for fitting of cumulative distribution. An example of the measured Gauss and Boltzman distributions obtained, using the three investigated measurement methods, is shown in **Figure 2** for the 10C? grade. Main differences between the Boltzman distributions obtained with the LI and IA – A analysis are discerned in the part of the curves corresponding to small grain size regions. These discrepancies may be explained due to the differences intrinsic to both measuring processes. In the LI method, any WC particle, even an extra coarse grain, can be crossed in its corner leading

to a really small length measured value. However, this is never the case in the IA process, where an equivalent length is determined for a given grain surface. This event is clearly discerned in the cumulative grain size distribution curves shown in **Figure 2**. On the other hand, the distributions determined with the IA - B method appear slightly shifted to higher values, due to the fact that several grain boundaries are not effectively recognized.

Figure 2. Comparison of the relative and cumulative distributions determined following the linear interception and image analysis studied methods for the investigated 11M? hardmetal grade.

The possibility of finding a correlation among grain size distributions obtained with the different studied measurement methods was also investigated. With that purpose, the d_{10} to d_{90} grain size values were subtracted from the Boltzmann cumulative distributions for all studied materials and measuring techniques by selecting a 10% interval between cumulative frequency values (i.e. the d_{10}, d_{20}, ..., d_{90} grain size values were selected). Subsequently, a linear correlation was established for each selected cumulative distribution probability between the grain size values determined using LI and IA techniques. The results of such analysis are given in **Figure 3**. As already observed in the Boltzmann distribution curves, determined grain size values using LI method are slightly higher than those values determined with IA techniques for low cumulative frequencies. Such trend changes progressively and is completely shifted at high cumulative frequencies when comparing LI and IA – A measurement methods. On the other hand, similar grain size values are obtained with both techniques in the medium frequency range. Regarding the IA – B technique, a similar evolution is observed, but determined grain size values are always slightly higher than those determined following the IA – A technique.

Figure 3. Comparison between carbide grain size values deduced from the cumulative distributions corresponding to the linear interception and image analysis techniques for different cumulative distribution percentages.

3.2. Assessment of contiguity of the carbide phase

As already mentioned in Section 1, the carbides phase contiguity is a key two-phase microstructural parameter to describe the correlation between microstructure and mechanical properties of cemented carbides. Following Roebuck and Bennett's approach [17], an extensive contiguity data collection from literature was here conducted [17,20,23–33] and C_{WC} values were plotted as a function of binder volume content (**Figure 4**). It was found that gathered data follows a similar trend to that described in previous works [1,17]. However, in this case the best fitting ($R^2 = 0.45$) was obtained for n and D values of 0.52 and 0.16 respectively. In any case, as previously referred and here inferred by the quite low best-fit R^2 value, a significant contiguity scatter was observed for a given binder content. Possible reasons for this large contiguity dispersion are the grain size influence as well as discrepancies associated with identification and definition of grain boundaries [1,17].

Figure 4. Contiguity dependence on the binder volume content (data collection from literature [17,20,23–33]). Full lines correspond to the best fitting curves proposed in pervious works (red line) [1,17] and in this investigation (green line).

OJO: correct numbering of references in Figure's legend (Figure 4)

Aiming for simultaneous evaluation of the influence of V_{binder} and d_{WC} on C_{WC}, collected data was plotted considering binder volume and carbide mean grain size as independent variables.

Results are shown as a 3D surface plot in **Figure 5**. Note that range values for mean grain size and binder volume content were defined as 0 - 2.5 μm and 5 - 35% respectively. Subsequently, a correlation was proposed between the obtained surface and a generic double exponential relation of type:

(4)

The best-fitting (R^2 = 0.85) was obtained when Eq. (4) constants take values of z_0 = 0.036, B = 0.973, C = 3.901 and D = 0.249.

Figure 5. C_{WC} values plotted as a function of mean grain size (d_{WC}) and binder volume content (V_{binder}) as independent variables.

Thus, a 3D colormap surface was drawn (**Figure 6**) according to best-fit determined values for Eq. (4). On the other hand, **Figure 7** shows the relation between C_{WC} and V_{binder} for different d_{WC} values. In accordance with literature (e.g. Refs. [17,20]), contiguity decreases when increasing binder content due to a higher probability of the binder phase to surround ceramic particles as the binder content becomes larger. As previously speculated [1,17], it is interesting to remark the dependence found between scatter of the contiguity and mean grain size of the carbide phase. Such dispersion becomes smaller as the binder content increases. Regarding the influence of mean grain size on contiguity of carbide phase, it should be highlighted the finding of: (1) an inverse relation between both parameters, and (2) less significant microstructural effects on contiguity for submicrometric grain sized grades.

Figure 6. C_{WC} values plotted as a function of d_{WC} and V_{binder}, as independent variables according to best fitting parameters determined for Eq. (4).

Figure 7. Contiguity as a function of the binder content for different mean grain size levels.

Finally, estimated contiguity values using Eq. (4) were plotted against those attained by following the linear intercept method and results are shown in **Figure 8**. An excellent concordance between them was found (R^2 = 0.91), validating then the proposed empirical relation as an adequate time-saving and efficient procedure for assessing contiguity in cemented carbides.

Figure 8. Linear dependence between the contiguity values experimentally determined following the linear interception technique and the values estimated according to the empirical relationship proposed in Eq. (4).

4. CONCLUSIONS

In this study an automatic image analysis protocol for grain size assessment in WC-Co cemented carbides is presented. A very accurate relation is found between the average grain size values determined following the linear intercept method and those attained with the image analysis protocol here described. Moreover, a method to correlate grain size distribution curves obtained with both measurement procedures is detailed. On the other hand, an empirical relation for contiguity determination in hardmetals is proposed. This relation is based on an extensive data collection from open literature, and considers the simultaneous effect of binder content and grain size on contiguity of the carbide phase. A quite satisfactory agreement is found between the grain size and contiguity values attained by means of the linear intercept method and those determined according to the two-stage methodology proposed in this study. This validates it as an effective time-saving process for microstructural characterization (grain size and contiguity of carbide phase) of WC-Co cemented carbides.

ACKNOWLEDGEMENTS

This work was financially supported by the Spanish Ministerio de Economía y Competitividad (Grant MAT2012-34602). J.M. Tarragó and D. Coureaux acknowledge Ph.D. scholarships received from the collaborative program Sandvik Hyperion / Universitat Politècnica de Catalunya and the Agencia Española de Cooperación Internacional (MAEC-AECID), respectively.

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