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SciVerse ScienceDirect
Acta Materialia 61 (2013) 4726–4733
Acta MATERIALIA
www.elsevier.com/locate/actamat

On the three-dimensional structure of WC grains in cemented carbides

Ida Borgh [a,*], Peter Hedström [a], Joakim Odqvist [a], Annika Borgenstam [a], John Ågren [a], Ali Gholinia [b], Bartlomiej Winiarski [b,c], Philip J. Withers [b], George E. Thompson [b], Ken Mingard [c], Mark G. Gee [c]

[a] *Department of Materials Science and Engineering, KTH Royal Institute of Technology, 100 44 Stockholm, Sweden*
[b] *School of Materials, University of Manchester, Manchester M13 9PL, UK*
[c] *Materials Division, National Physical Laboratory, Hampton Road, Teddington TW11 0LW, UK*

Received 22 February 2013; received in revised form 9 May 2013; accepted 10 May 2013
Available online 6 June 2013

Abstract

In the present work, the size distribution and shape of WC grains in cemented carbides (WC–Co), with different Co contents, have been investigated in three dimensions. Direct three-dimensional (3-D) measurements, using focused ion beam serial sectioning and electron backscattered diffraction (EBSD), were performed and a 3-D microstructure was reconstructed. These measurements were supplemented by two-dimensional (2-D) EBSD and scanning electron microscopy on extracted WC grains. The data from 2-D EBSD collected on planar sections were transformed to three dimensions using a recently developed statistical method based on an iterative inverse Saltykov procedure. This stereological analysis revealed that the assumed spherical shape of WC grains during the Saltykov method is reasonable and the estimated 3-D size distribution is qualitatively in good agreement with the actual distribution measured from 3-D EBSD. Although the spherical assumption is generally fair, the WC grains have both faceted and rounded surfaces. This is a consequence of the relatively low amount of liquid phase during sintering, which makes impingements significant. Furthermore, the observed terraced surface structure of some WC grains suggests that 2-D nucleation is the chief coarsening mechanism to consider.

Keywords: Three-dimensional EBSD; Three-dimensional characterisation; Carbides; Coarsening; Inverse Saltykov

1. Introduction

Cemented carbides are in effect metal matrix composite materials containing a high fraction of the ceramic phase. Traditionally, they comprise hard WC grains embedded in a Co-rich ductile binder phase. The WC grain size has a strong influence on the mechanical properties and, during liquid-phase sintering, the average WC grain size will increase due to coarsening. Some grains have a tendency to grow and become significantly larger than the average grain size through so-called abnormal grain growth. Since this phenomenon may seriously degrade the mechanical properties of cemented carbides, numerous studies have previously focused on understanding what controls abnormal grain growth, e.g. see Refs. [1–5].

Recently, Mannesson et al. [6] presented a new model for simulating the time evolution of WC grain size distributions. The model takes two-dimensional (2-D) nucleation, mass transfer across the interface and long-range diffusion into account. In order to simulate abnormal grain growth, the 2-D nucleation of new atomic layers is important since the coarsening of faceted WC grains is believed to be controlled by the 2-D nucleation [3].

An important input to the model by Mannesson et al. is the initial three-dimensional (3-D) grain size distribution. Hence, considerable work has been devoted to the development of stereological tools to evaluate 3-D parameters

* Corresponding author. Tel.: +46 8 7908340; fax: +46 8 100411.
E-mail address: iborgh@kth.se (I. Borgh).

1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2013.05.008

from 2-D data. Recently, Jeppsson et al. [7] presented a method for transforming 2-D size distributions to three dimensions. They called it the inverse Saltykov method and it uses a combination of statistical kernel estimators [8] and a Saltykov method [9].

In this context the difficulty of evaluating grain size distribution from 2-D observation is well known [10]. However, although the properties of the material must be derived from the 3-D structure, our current understanding of WC grain coarsening is based exclusively on 2-D experimental observations. The aim of the present work is thus to investigate the microstructure of cemented carbides by direct 3-D measurements. The measurements are performed using a combination of electron backscattered diffraction (EBSD) and focused ion beam (FIB) serial sectioning, and the 3-D structure is subsequently reconstructed. This method is hereafter referred to as 3-D EBSD. The direct 3-D measurements are compared with stereological conversions. The new data are used to both validate the stereological method, transforming 2-D to 3-D data, as well as to further develop the understanding of grain coarsening and previously developed theoretical models [6] describing the process.

Table 1
EBSD parameters.

	WC–6% Co alloy	WC–20% Co alloy
3-D EBSD		
Microscope	FEI Nova 600-I	FEI Nova 600-I
Electron current (nA)	10	10
FIB current (nA)	0.5	0.5
Acceleration voltage (kV)	30	30
Binning	4 × 4	4 × 4
Volume (voxels)	175 × 175 × 61	162 × 125 × 61
Volume (μm^3)	8.75 × 8.75 × 3.05	8.1 × 6.25 × 3.05
Detected phases	WC, Co(fcc), Co(hcp)	WC, Co(fcc), Co(hcp)
Step size in *x–y–z* (nm)	50	50
Grain boundary definition	3°	3°
Smallest grain definition	24 voxels in volume	24 voxels in volume
2-D EBSD		
Area (μm^2)	25 × 22	50 × 50
Detected phase	WC, Co(fcc), Co(hcp)	WC, Co(fcc), Co(hcp)
Step size (nm)	50	50
Grain boundary definition	3°	3°
Smallest grain definition	8 pixels in area	8 pixels in area

2. Experimental work

Two alloys with different Co contents, 6 and 20 mass%, respectively, and different initial powder-particle sizes were studied. The alloys were produced by conventional powder metallurgical methods. The sintered samples were metallographically prepared by mechanical polishing using 9 and 1 μm diamond suspension in consecutive steps. The final polishing of samples used for the 2-D EBSD was performed by ion polishing to remove the deformed surface layer. 2-D EBSD measurements were performed with a step size of 50 nm and three phases were included in the analysis, namely WC, Co(face-centred cubic, fcc), and Co(hexagonal close packed, hcp). Due to different initial grain sizes for the two alloys, different areas were analysed to achieve good grain statistics. An area of $25 \times 22\ \mu m^2$ was selected for the WC–6% Co alloy and an area of $50 \times 50\ \mu m^2$ was selected for the WC–20% Co alloy. Parameters for the 2-D EBSD analyses are summarised in Table 1. The HKL Channel 5 Tango software [11] was used for post-processing of the data. A noise reduction of non-indexed data points was performed for all three phases and the non-indexed data points were reduced from 3% to 1.5% for the WC–6% Co alloy and from 15% to 7.5% for the WC–20% Co alloy. In the present work, only the WC is considered; however, the evaluated phase fraction of Co is 9.5% for the WC–6% Co alloy (9.5% Co(fcc)) and 19.2% for the WC–20% Co alloy (19.0% Co(fcc) and 0.2% Co(hcp)). It should be noted that since the Co is ductile compared to the WC it will be more affected during the sample preparation, which could lead to poor indexing of the Co. The WC–WC boundaries were defined as having a misorientation angle larger than 3°, well above the measurement error (~0.5°). This rather small misorientation value was selected to assure that low-angle boundaries were also detected. The smallest detectable grain was defined as having an area of eight pixels ($0.02\ \mu m^2$) and grains touching the edges of the micrograph were excluded from the analyses. The grain size was evaluated by representing the grains with an equivalent circle diameter.

The 3-D size distribution was inferred from the 2-D sections using the inverse Saltykov method [7], hereafter referred to as the 3-D Saltykov method. The 2-D size distribution was first constructed using the kernel estimators, which circumvents the limitations of histograms [6], and presents the size distribution as a continuous function. Subsequently, the 3-D size distribution is optimised from the smoothed 2-D size distribution, using an iterative procedure assuming spherical grains. The number of mesh points used for the 3-D Saltykov was 500.

The samples for 3-D EBSD were carefully prepared to produce two polished and perpendicular surfaces. The 3-D EBSD was performed in a FEI Nova NanoLab 600-I instrument. A step size of 50 nm in the *x–y–z* directions was used and the same three phases as in the 2-D analysis were included. The analysed volume was $8.75 \times 8.75 \times 3.05\ \mu m^3$ for the WC–6% Co alloy, and $8.1 \times 6.25 \times 3.05\ \mu m^3$ for the WC–20% Co alloy. Parameters for the 3-D EBSD analyses are summarised in Table 1. More information on the 3-D EBSD technique can be found in Refs. [12–15]. The grain boundaries of WC–WC were defined as having a misorientation angle larger than 3° and the smallest detectable grains were defined as having a volume of 24 voxels ($0.003\ \mu m^3$). In order to improve the statistics for the 3-D EBSD analyses, regarding size distribution analysis, grains touching the boundaries of the