Computational Materials Science 93 (2014) 144–150

Contents lists available at ScienceDirect

Computational Materials Science

journal homepage: www.elsevier.com/locate/commatsci

Characterization and comparison of grain boundary character distributions in cemented carbides with different binder phases

Xiaokun Yuan [a,*], Chongbin Wei [b], Dongtao Zhang [a], Jie Zhu [c], Xuexu Gao [c]

[a] College of Materials Science and Engineering, Beijing University of Technology, Beijing 100124, China
[b] Zhuzhou Cemented Carbide Cutting Tools Co. Ltd., Zhuzhou 412007, China
[c] State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China

ARTICLE INFO

Article history:
Received 30 December 2013
Received in revised form 13 June 2014
Accepted 14 June 2014
Available online 19 July 2014

Keywords:
Electron backscattered diffraction
Five parameter analysis
Grain boundary character distribution
90°/[10−10] Grain boundary
Cemented carbide
Binder phase

ABSTRACT

The electron backscattered diffraction measurement, coupled with a statistical and stereological approach namely five parameter analysis method, was used to investigate the character distributions of carbide/carbide grain boundaries in a WC-6 wt%Co cemented carbide sample and a WC-6 wt%Ni cemented carbide sample. The 90°/[10−10] boundary was found as the most frequently occurring carbide/carbide boundary type in both samples, however, the populations as well as spatial distributions of the 90°/[10−10] boundary varied with different binder phases. Moreover, traces of the 90°/[10−10] grain boundaries may have separate configurations or adjoin at triple junctions. The geometry features of 90°/[10−10] boundary traces indicate that the energy equilibrium of the referred carbide grains has significant potential on the crack propagation.

1. Introduction

Cemented carbides generally comprise hard carbide phases (e.g. tungsten carbide) embedded in tough metallic matrixes, namely binder phases (e.g. cobalt [1], nickel [1] or copper [2]), and such composites have outstanding mechanical properties and are widely used where hardness and erosion/abrasion resistance are highly required. The mechanical properties of cemented carbides are strongly influenced by their dense polycrystalline microstructures, which can be described as irregularly shaped and approximately polygonal crystals joined at internal interfaces that are referred to as grain boundaries or phase boundaries [3], and characterization of the interfaces distribution is thus an important issue for understanding and improving the performances of the composites. In this field, grain boundary character distribution (GBCD) [4] is a significant aspect because the mesoscale structure of the grain boundary network influences the integrity and performances of the materials [5].

In measuring the GBCD within cemented carbides, one of the major concerns is the special carbide/carbide grain boundaries [6]. Among these boundaries, the 90°/[10−10] boundary, with the basic crystallographic feature of a 90° rotation about the [10−10] axis [7], and with low interfacial energy as well as high work of separation [8], is attracting special attention [6,9–13]. Note that the hexagonally close packed lattice structure of tungsten carbide has a c/a ratio of 0.976 (here c = 0.2837 nm and a = 0.2906 nm) [14] which is quite close to 1, so the 90°/[10−10] boundary can be referred to as $\Sigma 2$ boundary in coincidence site lattice (CSL) notation [7], where the low Σ value represents high reciprocal density of coinciding sites and symmetrical configuration of dense planes [15]. Among the GBCD measurement approaches, the electron backscattered diffraction (EBSD) technique is a highly capable tool that makes the collection of large data sets (and thus the GBCD measurement) a routine matter. During EBSD measurement, carbide/carbide boundary planes intersect the plane of EBSD observation, and boundary traces can therefore be consequently obtained and CSL boundaries can be routinely calibrated using Brandon's criterion [16]. In this case, distribution feature of boundary traces on two-dimensional planar section is an important proxy of GBCD in a three-dimensional sense. Recently, a statistical and stereological approach named "five parameter analysis (FPA)" has been developed to calculate the GBCD from EBSD data [17], and the validity of this method has been widely reported in a variety of materials [5]. Using this method, the GBCD is expressed in terms of five macroscopically observable parameters, including three Euler angles for describing the lattice misorientation across the boundary and two spherical angles for describing the orientation of the grain boundary plane normal. Thus, the FPA provides a

* Corresponding author. Tel./fax: +86 10 67396260.
E-mail address: yuanxiaokun@bjut.edu.cn (X. Yuan).

http://dx.doi.org/10.1016/j.commatsci.2014.06.022
0927-0256/

comprehensive description of the grain boundaries within a polycrystal.

The author's earlier works mainly concern different factors that could influence the GBCD in cemented carbides, and these factors may include plastic deformation [18], densification method [19], carbide grain size [20], binder fraction [21], and binder phase type. Binder phase type is thought to be an essential element in both structure development during sintering and crack evolution during application of cemented carbides [2,22]. Previous literatures mainly concern the role of the binder phases in the form of the mean free path (the distance separating carbide grains), and to a lesser extent, the contiguity between carbide or binder phases [1,23–25]; however, the impact of different binder phases on the GBCD within the cemented carbides has not been extensively investigated. Accordingly, the major purposes of present work are as follows: first, to preliminarily check whether the different binder phases can affect the GBCD; second, to investigate the population of special boundaries in cemented carbides; finally, to study the geometry feature of 90°/[10 −10] boundary traces and the possible connection to the performances of cemented carbides. The significance of current work is to reveal the applicability of FPA as a computational method to analyze the GBCD in cemented carbides with different binder phases; therefore, the GBCD is mainly concerned in current work, and the GBCD is analyzed by the combination of EBSD technique and FPA computational method. Note that the average carbide grain sizes in the samples were kept nominally the same, which makes the GBCD comparison meaningful.

2. Material treatment and data collecting

Two cemented carbide samples are selected in this work. The samples were prepared from the well-mixed tungsten carbide powder and binder phase powders, and the purity of these powders is higher than 99.9%. The starting tungsten carbide powder came from the same batch, with a grain size distribution mainly between 0.2 μm and 0.3 μm. The mixed powders were then sintered by a hot isostatic press (sinter-HIP) process in a 6 MPa argon atmosphere with a nominal sintering temperature of 1400 °C maintained for 30 min. Sample 1 (similarly hereinafter) has a cobalt fraction of 6 wt%, and sample 2 (similarly hereinafter) has a nickel fraction of 6 wt%. Both samples have an average carbide grain size of about 1 μm, and the samples are with no intentional alloying additions.

The samples were then treated by regular metallographic polishing method and afterwards were etched in Murakami's reagent (1 g potassium + 1 g sodium + 10 ml distilled water) for no more than 5 s. These treatments yielded sample surfaces suitable for EBSD mapping. The EBSD measurements were then performed by a high speed EBSD detector incorporated in a Zeiss Supra 55 scanning electron microscope. To ensure the accuracy of the measurements, the data were recorded with a step size of 0.05 μm.

The obtained EBSD data sets were then exported to EDAX OIM Analysis 5.31 software for analyzing. The first step in EBSD data processing was to use a level four cleanup procedure to correct spurious points in the orientation map due to incorrect indexing, that is, each measured orientation pixel was calibrated by being compared with the orientation of at least four nearest neighbors. Afterwards, the microstructures of the samples were indicated by the inverse pole figure (IPF) maps of carbide phases and IPF maps of binder phases. Carbide grain contours and special carbide/carbide boundary traces were manually imposed on the IPF maps in grayscale mode, and some selected representative 90°/[10 −10] boundary traces were manually imposed on the image quality (IQ) maps. The carbide grain size distribution was measured by the linear intercept method and was represented in diameter. The Brandon criterion [16] was used to determine the fraction of special carbide/carbide boundaries.

3. GBCD calculation

In current work, the GBCD of carbide/carbide boundaries is calculated in the following steps:

First, the orientation relationship between carbide grains was revealed by the misorientation angle distribution, and note that such distribution is featured by a single misorientation angle parameter; therefore, each boundary is differentiated by its misorientation angle, while the misorientation axis is not considered [5].

Then, the orientation texture of carbide/carbide boundaries was characterized by the misorientation distribution function plotted in the axis-angle space, namely axis/angle MDF, which is calculated by the harmonic series expansion method, and the method expands the misorientation distribution function into a series of generalized spherical harmonics. When both axis and angle parameter of the misorientation are considered, each boundary can then be described by three independent parameters, two for the rotation axis and one for the rotation angle; therefore, each position in the axis/angle MDF corresponds to a certain misorientation type [5].

The FPA method presents a more comprehensive description of the GBCD, and the principle of such method is to evaluate the GBCD by virtue of five independent parameters, that is, three Euler angles describing the lattice misorientation (abbreviated as Δg) across the boundary, and two spherical angles describing the orientation of the grain boundary plane normal (abbreviated as n); therefore, the five-parameter GBCD can then be parameterized as the function of $\lambda(\Delta g,n)$, which represents the relative occurring frequency of a certain grain boundary type distinguished by Δg and n within a polycrystal. Note that a set of Δg and n corresponds to a certain CSL boundary type with associated angle/axis combination, and therefore, for the selected 90°/[10 −10] boundary in current work, the orientation texture of its boundary plane can then be represented by the corresponding grain boundary plane orientation distribution function (GBP-ODF), and the result is presented by a stereographic projection.

In OIM Analysis software, the GBP-ODF is the integrated version of the FPA method and is developed under the auspices of the (former) MRSEC at Carnegie Mellon University. See the FPA programs at the webpage of reference [26] and see detailed algorithm description in reference [6,9,17]. Note that in this work, both axis/angle MDFs and GBP-ODFs are plotted in units of multiples of a random distribution (MRD). Moreover, the GBP-ODF needs a sufficient number of boundary traces within the plane section (2×10^5 boundary traces for hexagonal symmetry). Therefore, the orientation maps need to be merged together to meet this requirement.

4. Results

Orientation maps of the sample 1 are shown in Fig. 1, in which the individual WC grains are distinguished according to the hexagonal orientation coloring legend, with cobalt phase indexed as dark regions (see Fig. 1a), and the cobalt grains are distinguished by the hexagonal orientation coloring legend, with carbide grains indexed as dark regions (see Fig. 1b). Similarly, orientation maps of the sample 2 are presented in Fig. 2, where the individual WC grains are determined according to the hexagonal orientation coloring legend, with nickel phase indexed as dark regions (see Fig. 1a), and the nickel grains are discriminated by the cubic orientation coloring legend however, with carbide grains indexed as dark regions (see Fig. 2b). Both Figs. 1 and 2 exhibit the microstructures