Measurement of WC Grain Size in Nanocrystalline WC-10Co Hardmetal

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Abstract

The linear intercept (LI) method was used to quantitatively measure the intercepts of WC grains in nano-grained WC-10Co hardmetal. When the surveyed intercept numbers of WC grain exceeded 200, the statistic data for the mean grain size of WC were reproduced. The discriminative minimal grain size of used LI method was 12 nm; the maximum intercept of WC grain was 109 nm; the average intercept of WC grains was 45 nm and the corresponding 3D mean grain size of WC was 70 nm which is agreeable with the XRD outcome.

Keywords: nanocrystalline, hardmetal, grain, measurement

1. Introduction

There are two standard ways to determine the average grain size in metals [1, 2]. One is linear intercept (LI) method; the other is equivalent circle diameter (ECD) method. The LI method is a preferred standard for estimating the grain size of WC in hardmetals [3-5].

In recent years, some researches on the effects of doping a new invented VC-based multi-grain-growth-inhibitor on the fabricating processing, microstructure and properties of ultrafine and nanocrystalline WC-Co hardmetals have been conducted by the authors. The fully densified WC-10Co (in mass%) hardmetal can be produced by favorable technical processing. The aim of the present work is to evaluating the grain size of WC in the alloy.

2. Experimental and Results

The vertical cross section of the sintered cemented carbide was carefully cut, polished and etched in Murakami reagent separately. The well-defined second electron (SE) images for the microstructure of WC-10Co alloys were obtained by high resolution Philips XL30 S-FEG field emission scanning election microscope (FESEM) when magnification was equal or higher than 100,000:

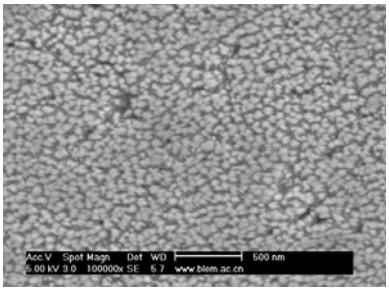

Fig. 1. Typical SE image of nano-grained WC-10Co alloy by FESEM

According to the counted number of WC grains, 3 to 10 mages were taken at different positions on the samples. The lengths of WC grains (l) were measured on a randomly settled testing line by the length detection and calculation function of a picture analysis software. The measured lengths should be transferred to its rear intercepts (I) by the formula as follows:

$$I = \frac{l}{L} \times M \qquad (1)$$

In which: L-measured length symbol of FESEM image; M-nominal length of length symbol.

The distribution of WC grains as the results of quantitative analysis for 1162 grains of WC was demonstrated. The effects of deviations in the testing results due to differences in visual perception, judgments, and operation skills for different operator are summarized. Although different picture analyzing softwares were used, the skilled operator can reproduce the similar results with a deviation of ±5 nm. Nevertheless, when different operators use the same analyzing software, the errors for the average as well as maximum intercept of WC grain can increase to 10~15 nm. When the surveyed intercept numbers of WC grain exceeded 200, the statistic data for the mean grain size of WC were reproduced. The discriminative minimal grain size of used LI method was 12 nm; the maximum intercept of WC grain was 109 nm; the average intercept of WC grains was 45 nm and the corresponding 3D mean grain size of WC[6] was 70 nm.

The average grain sizes of WC were also determined by X-ray diffractor. The full width half maximum (FWHM) of smoothed XRD peaks and Bragg angle 2θ were detected. The relationship between average grain size of WC d and the Bragg angle θ can be expressed by the Scherer formula:

$$d = \frac{0.89\,\lambda}{B_{1/2} COS\ \theta} \qquad (2)$$

$$B_{1/2}=FWHM–FWHMs \quad (3)$$

In which: $B_{1/2}$-FWHM changes are affected only by the grain size finalization;

FWHWs-FWHM changes resulting from instrumental broadening that can be checked by

In order to increase the accuracy of XRD analysis, the diffraction spectrum within low angle range ($2\theta \leq 50^{o}$) was investigated[7]. The average grain size of WC was evaluated as the average for the $(001)_{WC}$, $(100)_{WC}$ and $(101)_{WC}$ peaks. The verified average grain size of WC is 94 nm which is quit close to the results of metallographic analyses.

3. Summary

1. With suitable metallographic sample making and Murakami etching condition, SE images of nanocrystalline WC-10Co hardmetals with decent contrast and clear profile of WC grains can be obtained by FESEM under 100,000 magnifications.
2. The linear intercepts of the WC grains are measured on the SE images. When the surveyed intercept numbers of WC grain on randomly settled testing line exceeds 200, the statistic datum for the mean grain size of WC can be reproduced.
3. The discriminative minimal intercept of WC grains was 12 nm. Although different picture-analyzing software was used, the skilled operator could obtain similar results with a deviation of ±5 nm. When different operators use the same analyzing software, the deviations increased to 10~15 nm.
4. Nano-grained WC-10Co hardmetals can be made by doping a VC-based multi-grain-growth-inhibitor and suitable technical process. The average intercept of WC grains is 45 nm and the corresponding 3D mean grain size of WC is about 70 nm which is consistent with the XRD results (94 nm) deduced from the Scherrer analysis.

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