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Tungsten carbide platelet-containing cemented carbide with yttrium containing dispersed phase

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Abstract: A fine and platelet tungsten carbide patterned structure with fine yttrium containing dispersed phase was observed in liquid phase sintered WC-20%Co-1%Y_2O_3 cemented carbide with ultrafine tungsten carbide and nano yttrium oxide as starting materials. By comparing the microstructures of the alloy prepared by hot-press at the temperature below the eutectic melting temperature and by conventional liquid phase sintering, it is shown that hexagonal and truncated trigonal plate-like WC grains are formed through the mechanism of dissolution-precipitation (recrystallization) at the stage of liquid phase sintering. Yttrium in the addition form of oxide exhibits good ability in inhibiting the discontinuous or inhomogeneous WC grain growth in the alloy at the stage of solid phase sintering.

Key words: cemented carbide; platelet tungsten carbide grain; grain growth inhibition; yttrium; sintering

1 Introduction

The concept of whisker and platelet reinforcement is well known in the field of advanced ceramics with improved toughness[1]. It has been demonstrated that this idea can be applied successfully as well to WC-based cemented carbide. Trigonal or truncated trigonal WC platelets were shown to form in-situ by reaction sintering processes or recrystallization[2−5]. Compared with the conventional platelet-free cemented carbide, optimized platelet-containing cemented carbide showed superior hardness and toughness combinations, improved wear and impact resistance, improved high-temperature creep resistance[2−3,5]. In this work, hexagonal plate-like WC grains were formed in situ through the mechanism of dissolution-precipitation (recrystallization) at the stage of liquid phase sintering of WC based cemented carbide, and inhomogeneous grain growth which is a very vexing problem in cemented carbide with ultrafine or nano tungsten carbide as starting material during the early-stage densification was suppressed by the addition of yttrium oxide.

2 Experimental

2.1 Starting materials

Tungsten carbide powder with FSSS (Fisher subsieve sizer) of 0.51 μm and total carbon content of 6.09% (mass fraction), and cobalt powder with FSSS of 0.93 μm and Y_2O_3 powder, 20 nm in single particle determined by BET specific surface area, were used as the starting materials of WC-20%Co-1%Y_2O_3 (mass fraction) cemented carbide.

2.2 Alloy preparation

As it is reported before[6−7], high efficiency of grinding and dispersion during ball-milling can be achieved via the surfactant effect of multicomponent organic medium and the particle-stabilization effects of surfactant and macromolecule. Based on the principle mentioned above, a new cemented carbide slurry preparation technique was developed. Tungsten carbide powder, cobalt powder, Y_2O_3 powder and paraffin wax

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based macromolecule compound were wet milled in multicomponent organic medium for 72 h with mass ratio of milling media (cemented carbide ball) to powder of 6:1. The liquid medium in the colloidal slurry was evaporated under vacuum.

Hot-press at the temperature of 1 260 ℃ for 60 min which is below the eutectic temperature was carried out by uniaxial hot press under vacuum. Liquid phase sintering was carried out at the temperature of 1 470 ℃ for 90 min in a conventional vacuum sintering furnace.

2.3 Microstructure observation and analysis

Polished sample was first observed by Olympus PMG3 optical microscope(OM) to check if there exists graphite phase, then was etched in sequence by: 1) the mixture of 10% KOH (mass fraction) and 10% $K_3Fe(CN)_6$ with equal volume for 5−10 s to detect whether η phase exists; 2) the mixture of 20% KOH and 20% $K_3Fe(CN)_6$ with equal volume for 3−4 min; 3) the saturated solution of $FeCl_3$ in concentrated HCl for 20−30 s to give good contrast between WC grains and the binder.

For stereographic observation of WC grains, the WC grains in the sintered compacts were extracted by removing the cobalt matrix with boiling hydrochloric solution. Cobalt residue was removed by dilute hydrogen peroxide solution and the extracted WC grains were rinsed sequentially with distilled water and acetone.

JEOL JSM-6360 LV scanning electron microscope (SEM) was used in the observation of the microstructure of the extracted WC grains. EDAX Genesis 2000 energy dispersive X-ray spectroscopy(EDXS) was used in the qualitative analysis of the dispersed phase in the alloy.

3 Results and discussion

3.1 Microstructure and dispersed phase analysis

The microstructures of WC-20%Co-1%Y_2O_3 cemented carbide prepared by hot-press and by conventional liquid phase sintering are given in Fig.1 and Fig.2, respectively. Neither graphite phase nor η phase was observed in the alloy. It can be observed from Fig.1 that WC grains in the alloy by hot-press below the eutectic temperature are fine and homogenously distributed. Abnormal grain growth which is a very vexing problem in cemented carbide with ultrafine or nano tungsten carbide as starting material[8−10] was not observed. Accordingly, Y_2O_3 exhibits good ability in inhibiting both the continuous and discontinuous WC grain growth at the solid phase sintering stage. Nevertheless, a fine and platelet tungsten carbide patterned structure was observed in the liquid phase sintered sample. The maximum length of the platelet WC grains is as large as 10 μm, shown in Fig.2. Obviously, the ability of Y_2O_3 to inhibit the continuous grain growth can still remain to a considerable extent; however, it becomes ineffective in controlling the preferential grain growth at the liquid phase sintering stage.

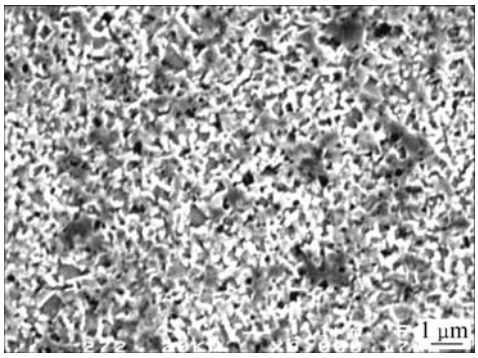

Fig.1 SEM microstructure of WC-20%Co-1%Y_2O_3 cemented carbide by hot-press below eutectic temperature (not etched by saturated solution of $FeCl_3$ in concentrated HCl)

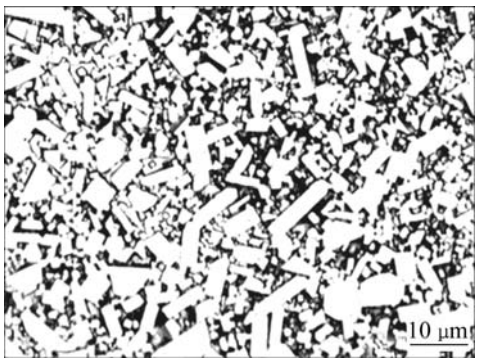

Fig.2 OM microstructure of WC-20%Co-1%Y_2O_3 cemented carbide by liquid phase sintering (additionally etched by saturated solution of $FeCl_3$ in concentrated HCl)

Liquid phase sintered sample which was not etched by saturated solution of $FeCl_3$ in concentrated HCl was observed by OM and SEM. The microstructure is shown in Fig.3. Along with WC and the binder phase, a fine dispersed phase (the dark one) with an average grain size less than 1 μm is observed. This dispersed phase cannot be identified if the binder is etched by saturated solution of $FeCl_3$ in concentrated HCl as shown in Fig.2, and has not been detected in the solid phase sintered sample. EDX spectra of the dispersed phase in three different positions indicated by the arrows in Fig.3(b) are given in Fig.4. Y, O, W, Co and C are detected in all the positions. Based on EDXS analysis results and the fact that although the addition amount of Y_2O_3 in the alloy is only 1%, the dispersed phase is in considerable amount, it can be deduced that yttrium has a very strong ability to combine the elements in the alloy to form a new phase during the liquid phase sintering process. Considering