



Determination of microstructure and mechanical properties of VC/Cr₃C₂ reinforced functionally graded WC-TiC-Al₂O₃ micro-nano composite tool materials via two-step sintering



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ABSTRACT

The effect of VC, Cr₃C₂ and combination (Cr₃C₂ and VC with varied weight ratios of Cr₃C₂/(Cr₃C₂+VC)) on the microstructure and mechanical properties of functionally graded WC-Al₂O₃-TiC composite synthesized employing two-step hot-pressing sintering (heated to 1700 °C and then immediately cooled to 1600 °C with a soaking time of 30min) was comprehensively investigated. Combined addition of VC and Cr₃C₂ with an appropriate ratio to WC-TiC-Al₂O₃ composite performed a more pronounced effectiveness on inhibiting grain growth and improving the homogeneous dispersion of Al₂O₃ nano-particulates in WC matrix than single addition of VC or Cr₃C₂ did. Furthermore, single Cr₃C₂ addition outperformed VC in grain refinement and uniform distribution of Al₂O₃. The experimental results show that excellent mechanical properties are achieved for Cr₃C₂-VC additions with Cr₃C₂/(Cr₃C₂+VC) weight ratio 0.6 with a hardness of 25.64 GPa, a flexural strength of 1209.6 MPa, a fracture toughness of 11.49 MPa mm^{1/2} and a surface residual stress of −591.9 MPa. High surface compressive stress, strong interface bonding, crack branching and bridging, crack deflection, microcracking and Al₂O₃ pullout are the major mechanisms contributing to the drastically enhanced flexural strength and fracture toughness. The five-symmetric-layer graded ceramics developed in this paper provide an exceptional combination of high toughness and high hardness, which are conducive to the application as metal cutting tools.

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1. Introduction

Binderless WC-based composites have been widely applied in metal cutting, mechanical seals and wear resistant parts due to their considerable hardness, strength and excellent corrosion and oxidation resistance [1–3]. However, it is difficult to consolidate pure WC free metallic binders to a full density and WC abnormal grain growth and the formation of sub-carbide W₂C is also unavoidable [4,5]. Binderless WC-based composites usually have such disadvantage as unsatisfactory flexural strength and fracture toughness [6].

In recent years, due to its industrial essentiality, sustained research efforts have been devoted to fabricating a fully dense binderless tungsten carbide composite featuring both considerable

hardness and excellent fracture toughness. Appropriate addition of carbides is contributed to the densification of WC-based ceramics [7], while the addition of cubic phases as TiC can improve the hardness of WC based composite [8]. The addition of Al₂O₃ to WC ceramics results in an improvement in the fracture toughness for the reason that Al₂O₃ additive facilitated the sintering and suppressed the formation of W₂C [9,10].

Many studies have proved that Hall-Petch-like relationship is applied to polycrystalline WC and the effective inhibition of grain growth is crucial for the hardness improvement of WC polycrystalline ceramics [11]. The addition of grain growth inhibitors, such as chromium carbide (Cr₃C₂) and vanadium carbide (VC), are considered as the most feasible approach to inhibit WC grain growth. In case of WC-Co cemented carbide, the relative effectiveness of inhibitors follow the order VC > Cr₃C₂ > NbC > TaC, and the inhibition mechanism is related to the solution-reprecipitation of WC grain in liquid Co phases [12]. Furthermore, combined addition of grain growth inhibitors with an appropriate ratio, such

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as VC and Cr_3C_2 , can perform a higher inhibiting effectiveness than single inhibitor addition [13,14]. With regard to binderless carbides such as WC–SiC and WC–SiC– Mo_2C ceramics, addition of grain growth inhibitors is also an effective method to markedly improve the mechanical properties [15–18], whereas literature on the effect of combined addition of grain growth inhibitors on WC–TiC– Al_2O_3 compacts are rather limited. On the other hand, the grain growth may well be inhibited to some degree by employing suitable sintering methods at relatively lower temperature and/or higher sintering rate [19], such as in two-step hot-pressing employing with external pressure [20], spark plasma sintering [21] or plasma pressure compaction [22].

Two-step sintering (TSS), put forward by Chen and Wang [23] as a promising approach to obtain high dense final sintered compact with nano-sized grains, has been widely applied to plenty of materials such as nanoceramics. According to TSS, a temperature interval existing between grain boundaries migration start and densification occurs [24], the green body would be heated to a high temperature, known as the first step effectively suppressing the grain growth, and then immediately cooled to a lower temperature with a long soaking time, referred to as the second step contributing much to the densification of materials. It is suggested that TSS can effectively improve the distribution of toughening particle and then enhance the toughness of materials [25,26]. Furthermore, WC may be more easier decomposed into W_2C in high sintering temperature compared with low temperature. TSS can inhibited the formation of W_2C to a certain extent [27].

In the present study, by employing two-step hot-press sintering, functionally graded WC–TiC– Al_2O_3 micro-nano composite tool materials with Cr_3C_2 –VC addition were fabricated with the auxiliary of dispersant (PVP and PEG). The effect of combined addition of Cr_3C_2 and VC with varied $\text{Cr}_3\text{C}_2/(\text{Cr}_3\text{C}_2+\text{VC})$ weight ratio on the microstructure and mechanical properties of the developed functionally graded WC–TiC– Al_2O_3 based composites was investigated.

2. Experimental procedure

2.1. Preparation

Powders of tungsten carbide (400 nm, 99.9% purity, Shanghai Chaowei nanotechnology Co. Ltd., China); Powders of titanium carbide (50 nm, 99.8% purity, Shanghai Chaowei nanotechnology Co. Ltd., China); Powders of vanadium carbide (80 nm, 99.9% purity, Shanghai Chaowei nanotechnology Co. Ltd., China); Powders of chromic carbide (100 nm, 99.9% purity, Shanghai Chaowei nanotechnology Co. Ltd., China); Powders of Aluminum Oxide (80 nm, 99.9% purity, Beijing Wangyong Technology Co. Ltd., China); Powders of PVPK-30 (99.9% purity, Shanghai Gobekie new material technology Co. Ltd., China); Powders of PEG (99.9% purity, Sino-pharm Chemical Reagent Co. Ltd., China) were used as starting materials. The compositions of powder mixtures for WC-based ceramics are given in Table 1.

In this paper, the starting materials were designedly ultrasonically dispersed and mixed in together at different milling stages. In the first stage, nanoparticles (Al_2O_3 , TiC, VC, Cr_3C_2) were ultrasonically dispersed by PVPK-30 and PEG in absolute alcohol for 60 min maintaining the temperature of 78 °C. After that, adding submicron WC powders into the mixture and ultrasonically dispersed for another 60 min. After dispersing, the mixed slurries were milled for 48 h in a high energy attrition mill with cemented carbide milling ball and absolute ethanol medium. The processing parameters of ball milling are demonstrated in Table 2.

Table 1

Compositions of powder mixtures for four designed samples (wt.%).

Composites	WC	Al_2O_3	TiC	Cr_3C_2	VC
Ceramics ASL	94	3	2	0	1.0
Ceramics AIL	89	6	4	0	1.0
Ceramics ACL	84	9	6	0	1.0
Ceramics BSL	94	3	2	0.2	0.8
Ceramics BIL	89	6	4	0.2	0.8
Ceramics BCL	84	9	6	0.2	0.8
Ceramics CSL	94	3	2	0.4	0.6
Ceramics CIL	89	6	4	0.4	0.6
Ceramics CCL	84	9	6	0.4	0.6
Ceramics DSL	94	3	2	0.6	0.4
Ceramics DIL	89	6	4	0.6	0.4
Ceramics DCL	84	9	6	0.6	0.4
Ceramics ESL	94	3	2	0.8	0.2
Ceramics EIL	89	6	4	0.8	0.2
Ceramics ECL	84	9	6	0.8	0.2
Ceramics FSL	94	3	2	1.0	0
Ceramics FIL	89	6	4	1.0	0
Ceramics FCL	84	9	6	1.0	0

SL: surface layer.

IL: interlayer.

CL: core layer.

2.2. Sintering and characterization

The powder mixtures were pressed into a circular die in the following order: surface layer mixtures, interlayer mixtures, core layer mixtures, interlayer mixtures and then surface layer mixtures. Finally, the composite powders were sintered by two-step hot-pressing in an inductive hot-pressing vacuum furnace (Model: ZRC85–25T, China). Fig. 1 illustrates the two-step sintering processes.

The flexural strength of the specimens was performed on a WDW-50E tester by using a three-point bending method with a 0.5 mm/min loading rate and a 14.5 mm span between the loading points. The hardness (HV_{10}) was measured employing a Vickers indenter (Model: MHVD-30AP, China) with a load of 98N and a duration time of 15 s using a diamond frustum of rectangular pyramid indenter with an opposite angle of 136°. The fracture toughness was defined by the crack length around the harness indent and the following equation was adopted [28,29]:

$$K_{IC} = 0.0889 \sqrt{\frac{HV \cdot P}{L}}$$

where L is the length of crack (mm), HV is vickers hardness (MPa), P is the load (N).

The microstructures of the samples were observed by a scanning electron microscope (SEM, QUANTAFEG 250, FEI Inc., USA) and the element distribution was investigated by energy dispersive spectroscopy (EDS, X-MAX30, Oxford Instruments Inc., UK). The phase identification of the specimens were analyzed by X-ray diffraction (XRD, D8ADVANCE, Bruker AXS Inc., Germany). The sintered samples were ground and polished by standard ceramographic methods and then etched in a Murakami's reagent consisting of $\text{Fe}_3[\text{K}(\text{CN})_6]$ (10 g), KOH (10 g) and distilled water (100 ml) for 5 min to expose the grain boundary of WC [30]. The average WC grain size was determined from SEM micrographs of the polished and etched samples surfaces employing linear intercept method. For each specimen, five images were taken of the microstructure and ten line segments were assessed in each image. The surface residual stress was measured by X-stress (XSTRESS 3000×, G2R system, Stresstech Oy Inc., Finland).

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