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Fabrication, microstructure and mechanical properties of self-diffusion gradient cermet composite tool materials



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ABSTRACT

Self-diffusion gradient cermet composite tool materials were fabricated by vacuum hot pressing process at different temperatures (from 1450 °C to 1575 °C) for different holding times (from 20 min to 50 min) under 32 MPa. A new subsurface layer enriched with metal binder was formed due to self-diffusion driving force after sintering. The surface layer had a higher hardness, while the substrate had a high flexural strength, as well as the subsurface layer had a good interface bonding strength. The relationships among sintering processes, mechanical properties and microstructure were discussed. The experimental results showed that both the sintering temperature and holding time had a great influence on the flexural strength and fracture toughness, while a relatively small effect on the hardness. For the sintering parameters considered, it was revealed that the sintering temperature of 1500 °C and the holding time of 40 min were in the vicinity of optimum to yield the desired mechanical properties and fine microstructure. Both grain sizes and the rim phase of the Ti(C,N) increased and the Ni content in the substrate decreased when sintering temperature varied from 1450 °C to 1575 °C and holding time varied from 20 min to 50 min. Compared with Ti(C,N)-based cermet, the self-diffusion gradient cermet exhibited better comprehensive mechanical performance.

1. Introduction

Cermet is a structural material in which fine grains of ceramic hard phase embed in a network of metal binder [1]. Therefore, cermet has the optimal properties of both a ceramic, such as high wear resistance and hardness, and those of a metal, such as the ability to undergo plastic deformation. Metallic materials such as nickel (Ni), molybdenum (Mo) and cobalt (Co) are usually used as binder for boride, carbide or nitride-based cermets because of their good wettability [2]. Brides, carbides and nitrides of transition metals attract significant interest since they have a wide field of applications such as high-temperature structural materials and wear-resistance parts [3–5].

Ti(C,N) and TiB₂-based cermets are high performance cermets with high melting points, high hardness and good wear resistance. For these reasons, they can be used as wear resistance parts such as cutting tools. Ti(C,N)-based cermets are much competitive in both the price and the performance compared with the WC-based hardmetals, especially when they are used in high speed cutting and finishing operations [6]. Normally, Mo is added into Ti(C,N)-based cermet to improve the wettability between the hard phase and metal binder [7]. In addition, some carbides such as WC [8], NbC [9] and VC [10] are used to accelerate densification of the Ti(C,N)-based cermets, refine grains and enhance mechanical properties. Wang et al. [10] demonstrated that the addition of VC can inhibit grain growth of micrometer grade Ti(C,N)based cermets by reducing the solubility of Ti atom in the liquid binder phase. Ying et al. [11] fabricated a tough and strong cermet with a flexural strength of 2210 MPa and a toughness of 10.1 MPa $m^{1/2}$, but a low Vickers hardness of 14.7 GPa was obtained, which is not suitable as cutting tool materials. TiB₂-based cermets are more suitable as cutting tool materials because they have higher melting point and hardness than those of Ti(C,N)-based cermets. What's more, boride composite has self-lubricating effect because of the formation of an oxide lubricating film formed by dissolution of boride into other phases [12]. The self-lubricating effect would decrease wear rate of the material and improve its life. However, TiB2 has a tendency toward low flexural strength and low fracture toughness, which remains an obstacle to its more widespread application [4]. Furthermore, the relatively low self-diffusion coefficient and predominant covalent bonding of TiB2 make a negative influence on its sintering ability [13]. Wang et al. [14] demonstrated that nearly fully densed (99% density) monolithic TiB₂ compacts were obtained at a sintering temperature of 2173 K, but low flexural strength (558 MPa) and

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Table 1

Composition of self-diffusion gradient cermet composite tool materials.

Symbols	TiB ₂ -TiC	Ti(C ₇ ,N ₃)	VC	Ni	Мо	Remarks
S M	86	84	4	6 12	4 4	Surface layer Substrate

Table 2

Sintering temperatures and holding times of self-diffusion gradient cermet composite tool materials.

Sintering temperatures	Holding times
1450 °C, 1475 °C, 1500 °C, 1525 °C, 1550 °C, 1575 °C	30 min
1500 °C	20 min, 30 min, 40 min, 50 min

fracture toughness (5.7 MPa m^{1/2}) were obtained because grains grew quickly and irregularly at such a high sintering temperature. Many studies [15–17] used metal binders (Co, Ni and Mo) as sintering additives to decrease sintering temperature of TiB₂ composites and high density of TiB₂ composites was obtained.

The strength, hardness and toughness can be changed by varying the chemical compositions and fabrication parameters. However, an increase in flexural strength and toughness is normally associated with a decrease in hardness of the cermet composites. In order to solve the contradiction, we designed the composites named after the new style gradient composites with a gradient structure formed by a selfdiffusion progress during sintering. Before sintering, the gradient composites are comprised of a TiB2-TiC cermet surface layer and Ti(C,N)-based cermet substrate. During sintering, a new subsurface layer enriched with metal binder is formed by a self-diffusion driving force of the elements. Compared with Ti(C,N)-based non-gradient cermet materials, it is expected that the gradient composites so fabricated would possess gradient properties, i.e., high hardness and good wear-resistance on the surface layer, good fracture toughness on the subsurface layer, and good flexural strength on the substrate. The subsurface layer would also improve interface bonding strength and impede delamination between the surface layer and substrate.

The objective of the present work is to enhance the mechanical properties of the self-diffusion gradient cermet composites by varying sintering temperatures and holding times. In this paper, self-diffusion gradient cermet composite tool materials were fabricated by a vacuum hot pressing process at varied temperatures (from 1450 °C to 1575 °C) for different holding times (from 20 min to 50 min) under 32 MPa. The effects of sintering temperature and holding time on the grain morphologies and mechanical properties of the sintered samples were investigated.

2. Experimental work

The mixtures of TiB2-TiC powders (99.9% purity, 0.7 µm size) and $Ti(C_7N_3)$ powders (99.9% purity, 0.5 µm size) were used as the starting materials. The average grain sizes of VC, Mo and Ni powders used as sintering additives were approximately 100-300 nm. The composition of the starting materials is listed in Table 1. The self-diffusion gradient composite tool materials were prepared by five steps. Firstly, the starting TiB₂-TiC and Ti(C₇N₃) powders were separately ball-milled using WC balls and ethanol as the medium for 24 h, and then dried in a vacuum dry evaporator. Secondly, Mo, Ni and VC powders were added into the TiB₂-TiC powders as the surface laver, and the Mo. Ni and Co powders were added into the $Ti(C_7N_3)$ powder as the substrate. Subsequently, these two mixed powders were separately ball-milled for 24 h and dried. Thirdly, the two mixed powders were sieved using a 200-mesh sieve. Fourthly, the substrate powder with 2.7 mm thickness and the surface layer powder with 400 µm thickness were put into a graphite die layer by layer, and then the green body with three layers was compressed under 10 MPa for 5 min. Ti(C,N) mixed powder was used as substrate, while TiB2-TiC mixed powder was used as upper and lower surface layer. Finally, the green body was sintered by a hotpressing process at different temperatures (from 1450 °C to 1575 °C) for different holding times (from 20 min to 50 min) under 32 MPa. Table 2 lists the detailed sintering process parameters.

The sintered disks were cut into blocks with dimensions of 3 mm×4 mm×30 mm, and the surfaces and cross-sections were then ground and polished. The plane of 3 mm×30 mm was the cross sections of the samples, while the plane of 4 mm×30 mm was the surface of the surface layer. Afterwards, the surface layer thickness was about 200-230 µm. The flexural strength was measured using a threepoint bending tester (Model WDW-50E, Shidai, China) with a span of 20 mm and a loading velocity of 0.5 mm/min. The surface hardness and substrate hardness were measured using a Vickers hardness tester (HV₁, HVS-1000, Hugong, China) with a load of 9.8 N and a dwell time of 15 s. The fracture toughness was measured using a single edge notched beam (SENB) test with a span of 30 mm and a loading velocity of 0.5 mm/min. The notches were manufactured on the cross-sections of the samples with a width of 0.16 mm using a diamond wire saw. The microstructure of cross-sections and the fractured surfaces were observed using scanning electron microscopy (SEM, SUPRA55, Zeiss, Germany).

3. Results and discussion

3.1. Effects of sintering temperature

3.1.1. Microstructure

Fig. 1 shows SEM micrographs of the polished cross sections of the



Fig. 1. SEM micrographs of the polished cross sections of the samples sintered at 1500 °C for 30 min. Self-diffusion gradient composites are comprised of surface layer, subsurface layer and substrate. (a) The whole cross section (b) an partial enlarged cross section.

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