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ARTICLE

Effect of Granule Size and WC Content on Microstructure and Mechanical Properties of Double Structure Ti(C,N) Based Cermets

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Abstract: The effect of starting granule size and addition of WC on the microstructure and mechanical properties of double structure Ti(C,N) based cermets was investigated. Results show that the Ti(C,N) based cermet granules are homogeneously distributed in the matrix. By increasing the WC content, the amount of hard phases with a white core/grey rim and a coreless structure in the matrix increases. A new phase with a four-layer composite structure is found. With the increase of the granules size, the fracture toughness increases, while the transverse rupture strength (TRS) and hardness show an opposite trend. With the increase of WC content, the fracture toughness and TRS increases, while the hardness decreases. The higher fracture toughness of the double structure Ti(C,N) based cermets is mainly owing to the branching, bridging and deflection of the crack, the formation of micro-cracks near the tip of the main crack, and the pull-out effect of granules.

Key words: double structure Ti(C,N) based cermets; microstructure; mechanical properties; toughening mechanism

Due to their excellent combination of high hardness, strength, wear resistance, thermal conductivity and chemical stability, Ti(C,N)-based cermets as cutting tool materials are used for high-speed milling, semifinishing and finishing of carbon, low alloy and stainless steels[1-4], while the low toughness severely limits the use of such materials, especially in rough machining application. The approaches for improving the toughness of Ti(C,N) based cermets are mainly as follows. One approach is toughening through whiskers[5] and carbon nanotubes[6]. The resulting crack deflection and bridging effects of whiskers or carbon nanotubes can provide the main contributions to fracture toughness[5,6]. Another approach is that Ti(C,N) based cermets as well as Al_2O_3 matrix composite ceramic with a larger starting powder size of added ceramic phase can obtain higher fracture toughness[7-9]. Double structure cemented (DC) carbide is a dual composite composed of WC-Co composite granules and binder matrix, which is a novel approach to improve fracture toughness by designing composite microstructure[10-12]. One of the technological interests for hard materials is to obtain higher fracture toughness by composite structures. Double cemented carbide, composed of WC-Co granules and matrix (Co), has higher fracture toughness compared with commercial WC-Co cemented carbide, which can be used as oil well drill bit inserts[10-12]. So far, detailed reports on improving the fracture toughness of Ti(C,N) based cermets by designing a composite structure of Ti(C,N) based cermets has not been conducted. Double structure Ti(C,N) based cermet has more degrees of freedom for material design than commercial Ti(C,N) based cermet, including the granule composition, size, volume fraction, strength, toughness, and hardness as well as matrix composition, strength, toughness, and hardness. All these variables enable wide variations of Ti(C,N) based cermets in mechanical properties. Controlling the microstructure and properties of granules as well as matrix can obtain an excellent combination of properties. As a cutter material, the challenge is not only to improve the fracture toughness, but also to improve or preserve TRS and hardness. One kind of double structure Ti(C,N) based cermet was developed in this paper. In order to maintain the high TRS and hardness, some

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cobalt was replaced by WC in the experiment, whereas the composition of matrix was slightly different from that of the reported double structure cemented carbide[10-12]. The primary goal of this study was to determine the effects of granules size and the content of WC in matrix on the mechanical properties and microstructure of double structure Ti(C,N) based cermets.

1 Experiment

Commercially available TiC (40 nm), TiN (20 nm), Co (2.46 μm), C (3.25 μm), Mo (2.33 μm) and WC (1.14 μm) were used as starting powders. The nominal composition of Ti(C,N) based cermets granules is 72 wt%TiC (nm)-8wt%TiN (nm)-15wt%Co-4wt%Mo-1wt%C. Previous work has shown that the addition of 4wt% Mo to Ti(C,N)-Co-Ni system can obtain better mechanical properties[13]. Powder mixtures were milled with WC-Co balls (ball-to-powder mass ratio, 8:1) by a planetary ball mill in an ethanol bath for 24 h and then dried. Green compacts were prepared by pressing at a uniaxial pressure of 180 MPa and sintered at 1430 °C for 1 h in vacuum (0.02~0.1 Pa), and then smashed, and finally sieved. The average granule sizes of starting Ti(C, N)-based cermets granules obtained by the Mastersizer-2000 laser particle size analyzer (Malven, UK) were 17, 35 and 48 μm, as shown in Fig.1. The morphology of the above granules was observed by SEM, as shown in Fig. 2a~2c. The morphology of higher magnification of Fig.3a is examined as shown in Fig.3b, to better understand the formation of the microstructure of the cermet. It can be clearly seen that the surface of the starting coarse granules is full of whisker-like fine granules with different sizes, as shown in Fig.3b. These whisker-like granules were easily fractured from the surface of starting coarse granules in the later milling process, and even some starting coarse granules can be directly fractured into 3~8 finer granules. Double structure Ti(C,N) based cermets were fabricated by a powder metallurgy process. In order to research the effects of the granules size and the content of WC on the microstructure and mechanical properties of double structure Ti(C,N) based cermets, the nominal composition designed is given in Table 1. The powders of the starting granules, Co and WC were milled with WC-Co balls by a planetary ball mill (ball-to-powder mass ratio, 8:1) in an ethanol bath for 4 h and then dried. Green compacts were prepared by pressing at the uniaxial pressure of 180 MPa and sintered at 1400 °C for 1 h in vacuum (0.02~0.1 Pa) based on the differential scanning calorimetry (DSC) results of Fig.4, wherein the melting temperature is about 1380 °C.

The microstructure of polished specimens was observed by scanning electron microscope (SEM, Hitachi SU8020, JEOL, Japan and Quanta 400, FEG, American) in back-scattered electron (BSE) mode coupled with an energy-dispersive spectrometer. Phase identification was carried out by X-ray diffractometry (XRD) mode of X" Pert PRO (Holland Panalytical). The fractured surface morphology was observed by SEM (JSM-6490LV, JEOL, Japan) in secondary electron mode. TG and DSC analysis of cermet powders were performed by the NETZSCH equipment at a heating rate of 10 °C/min. Rockwell hardness (HRA) testing was carried out on a common Rockwell hardmeter. The Rockwell hardness (HRA) of the cermet used for preparing the starting granules of double structure Ti(C,N) based cermets is 92. Fracture toughness (K_{IC}) was tested by an indentation method under an indentation load of 30 kg, according to the formula proposed by Shetty et al [14]. Transverse rupture strength (TRS) testing was carried out at room temperature by a three-point bending method using a universal testing machine with a loading rate of 0.5 mm/min (span is 30 mm). The specimen size was 6 mm×6 mm×30 mm for the TRS testing.

2 Results and Discussion

2.1 Effect of granule size on the microstructure

The microstructure of the double structure Ti(C,N)-based cermets with different starting granule sizes was observed by SEM-BSE, as shown in Fig.5. It can be seen that the granules with dark color are homogeneously distributed within the matrix with white color in the SEM/BSE mode. The dark color region of the granule consisted mainly of Ti(C,N) and correlated well with the light elements, while the white color region of matrix consisted mainly of (Ti,W)(C,N) and was rich in heavy element W in the SEM/BSE mode. It can be seen from Fig.4a to 4c that the granule size becomes coarser. The larger the raw powders of Ti(C, N)-based cermet granules, the larger the Ti(C,N)- based cermet granules in the matrix after sintering.

It is denoted from Fig. 6a that the granules can be further divided into three types as coarse, middle and fine based on their sizes as shown by the white arrows. The coarse granule

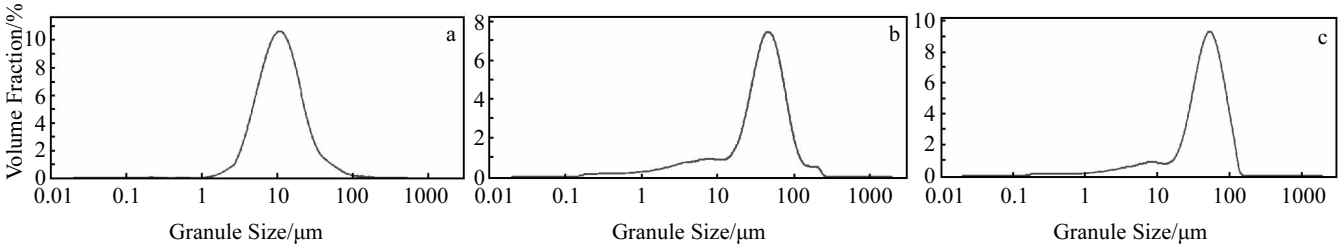

Fig.1 Granule size distribution of starting Ti(C,N) based cermet with different average granule sizes: (a) 17 μm, (b) 35 μm and (c) 48 μm

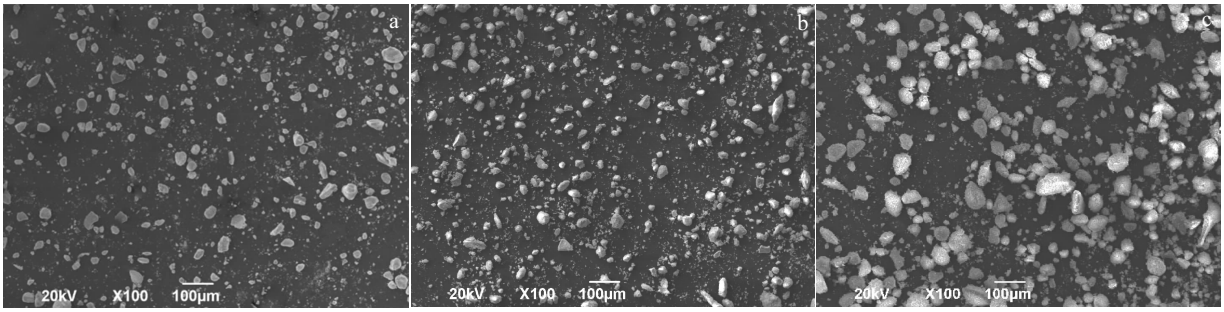

Fig.2 SEM images of the morphology of starting Ti(C,N) based cermet with different average granule sizes: (a) 17 μm, (b) 35 μm, and (c) 48 μm

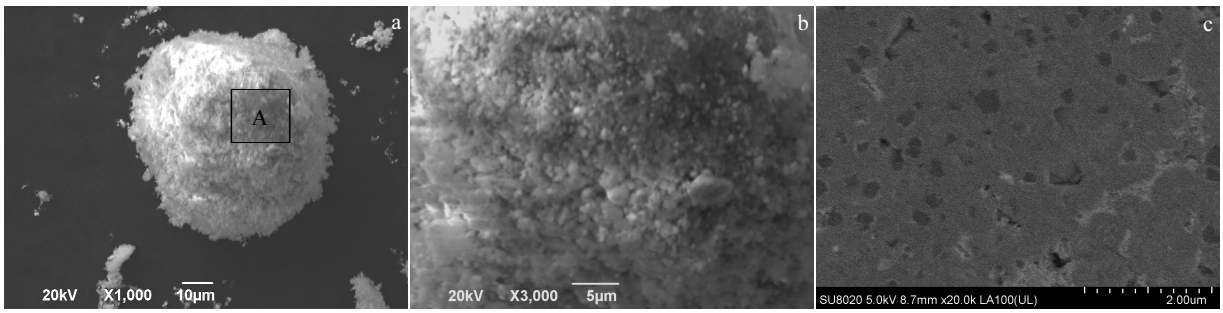

Fig.3 Single granule in Fig.2 (a), higher magnification of region A (b), and microstructure of starting granule (c)

Table 1 Nominal composition of the double Ti(C,N) based cermets

Cermet	Ti(C,N) average granule size/μm	Ti(C,N) content/wt%	Co content/wt%)	WC content/wt%
A	17	75	15	10
B	35	75	15	10
C	48	75	15	10
D	35	85	15	0
E	35	80	15	5
F	35	70	15	15
G	35	65	15	20

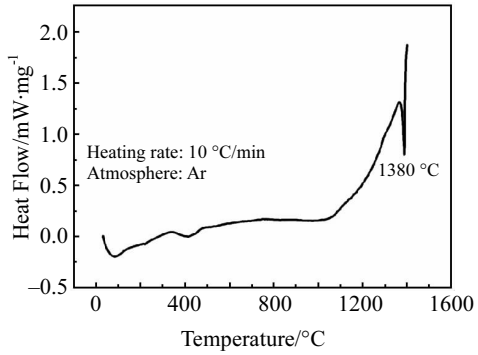

Fig.4 DSC curve of cermet B mixture powders

comes from the starting coarse granules remaining after sintering. The middle granule, with a size of 10~15 μm as shown by white arrows in Fig.6a, in the cermet should come from the remaining granules which are fractured from the starting coarse granule during the milling process. The fine granule with a size of 3~7 μm, as shown by the white arrow in Fig.6a, in the cermet should come from the fractured whisker-like fine granules on the surface of starting coarse granules, as shown in Fig. 3b. The higher magnification of zone A in Fig.6a denotes that the microstructure in the coarse granule is a typical dual phase, and the hard phase is a typical black core/grey rim or black core/white inner rim/grey outer rim structure, as shown in Fig.6b. The higher magnification of zone B in Fig.6a denotes that the hard phase nearby the coarse granule is similar to the microstructure in the coarse granule, as shown in Fig.6c. The results also denote that the microstructure of the middle and fine granules is the same as that of the coarse granule as shown in Fig.6a. The matrix is a hybrid composite microstructure of Co and hard phases including coreless, white core/grey rim and black core/white inner rim/

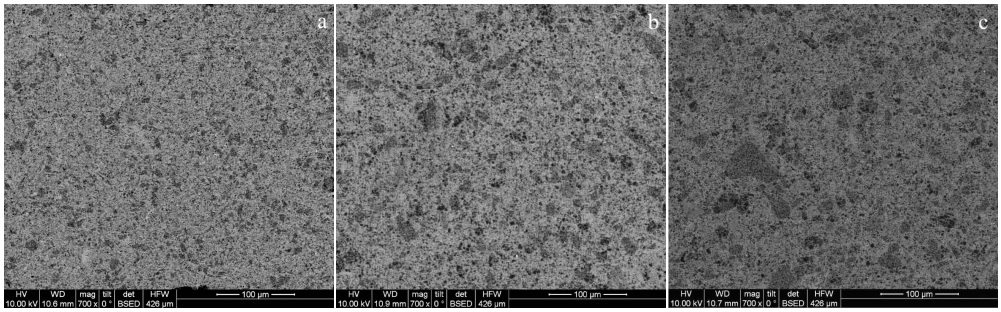

Fig.5 SEM/BSE images of cermets with different starting granule sizes: (a) 17 μm, (b) 35 μm, and (c) 48 μm

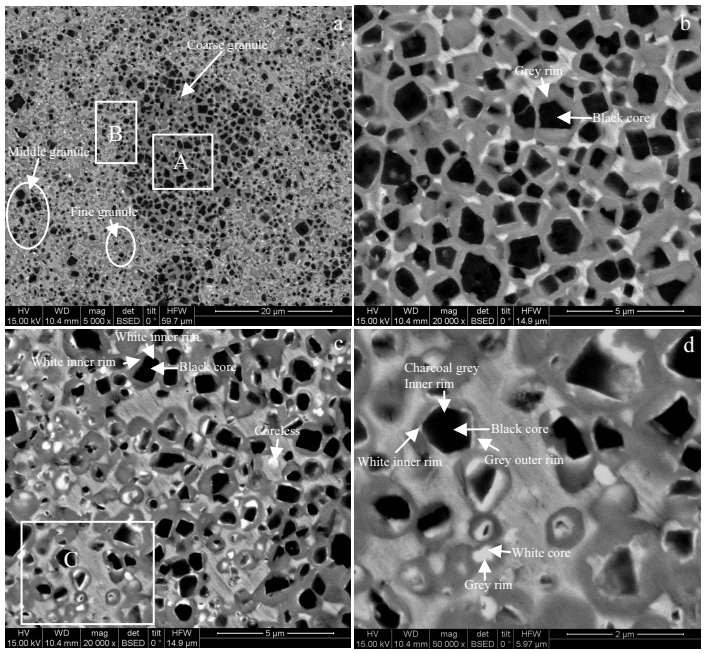

Fig.6 SEM/BSE images of cermet B (a) and corresponding zone A (b), zone B (c) in Fig.6a, and zone C (d) in Fig.6c

grey outer rim structures, as shown in Fig.6c and 6d, respectively. In addition, a new hard phase with a black core/charcoal grey inner rim/white inner rim/grey outer rim structure was found, as shown by the white arrows in Fig.6d. The higher magnification of zone C in Fig.6c denotes that the matrix is mainly hybrid composite structures of Co and hard phases including black core/grey rim, black core/white inner rim/grey outer rim, white core/grey rim and coreless structures, as shown by white arrows in Fig.6d.

In order to better understand the composition of hard phases of the double structure Ti(C,N) based cermets, SEM-EDS was employed for analysis of chemical compositions of hard phases in Fig.6, and the results are shown in Fig.7 and Table 2. The black core and grey rim in the typical black core/grey rim structure of hard phase in Fig.6b are TiC or Ti(C,N) and (Ti, Mo)C or (Ti,Mo)(C,N), respectively, based on the SEM-EDS results of Fig.7a and 7b and Table 2. The black core in black core/white inner rim/grey outer rim structure in the matrix of Fig.6c is also TiC or Ti(C,N), while the white inner rim is (Ti, W,Mo)C or (Ti,W,Mo)(C,N), because it is rich in W and Mo elements, and the grey outer rim is also (Ti,W,Mo)C or (Ti,W, Mo)(C, N) which has lower concentration of W and Mo elements than white inner rim based on the SEM-EDS results of Fig.7c~7e and Table 2, and agrees these results well with the previous researches[15,16]. The coreless and white core/grey rim structures of the hard phase in the matrix shown in Figs.6c and 6d are also found to be (Ti,W,Mo)C or (Ti,W,Mo)(C,N), respectively, based on the SEM-EDS results of Fig.7f to 7h and Table 2, which have been reported in previous research works[15-17]. In order to better understand the composition of the matrix of double structure Ti(C,N) based cermets, the energy spectrum of map scanning was used for analyzing the main elements Ti, W, Mo, Co and C, and the results are shown in Fig.8, It is indicated that the white core and coreless structure also have higher W content.

The formation mechanisms of hard phases have been

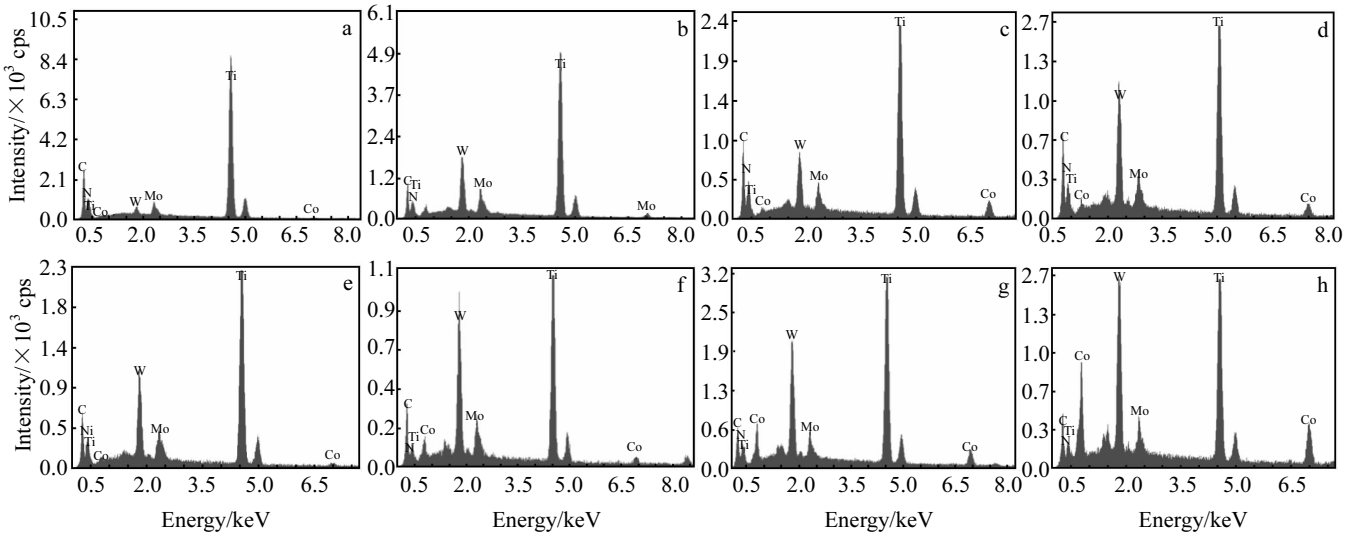

Fig.7 EDS spectra of various hard phases in Fig.6: (a) black core in the granule, (b) grey rim in the granule, (c) black core in the matrix, (d) white inner rim in the matrix, (e) grey outer rim in the matrix, (f) white core in the matrix, (g) grey rim around white core in the matrix, and (h) coreless structure

Table 2 EDS results of the microstructure in Fig.6 (at%)

Element	Granule		Matrix					
	Black core	Grey rim	Black core	White inner rim	Grey outer rim	White core	Grey rim	Coreless
Ti	53.05	52.28	42.90	38.94	47.51	39.36	40.39	33.26
W	0.81	3.53	03.55	06.68	05.96	9.06	07.06	09.79
Mo	0.86	2.09	01.07	01.52	01.71	1.55	01.41	01.31
C	38.75	30.59	40.46	41.26	34.67	38.35	33.94	27.34
N	6.32	3.95	07.53	06.69	05.89	0.00	01.25	00.00
Co	0.20	7.55	04.49	04.90	4.25	11.58	15.96	28.30

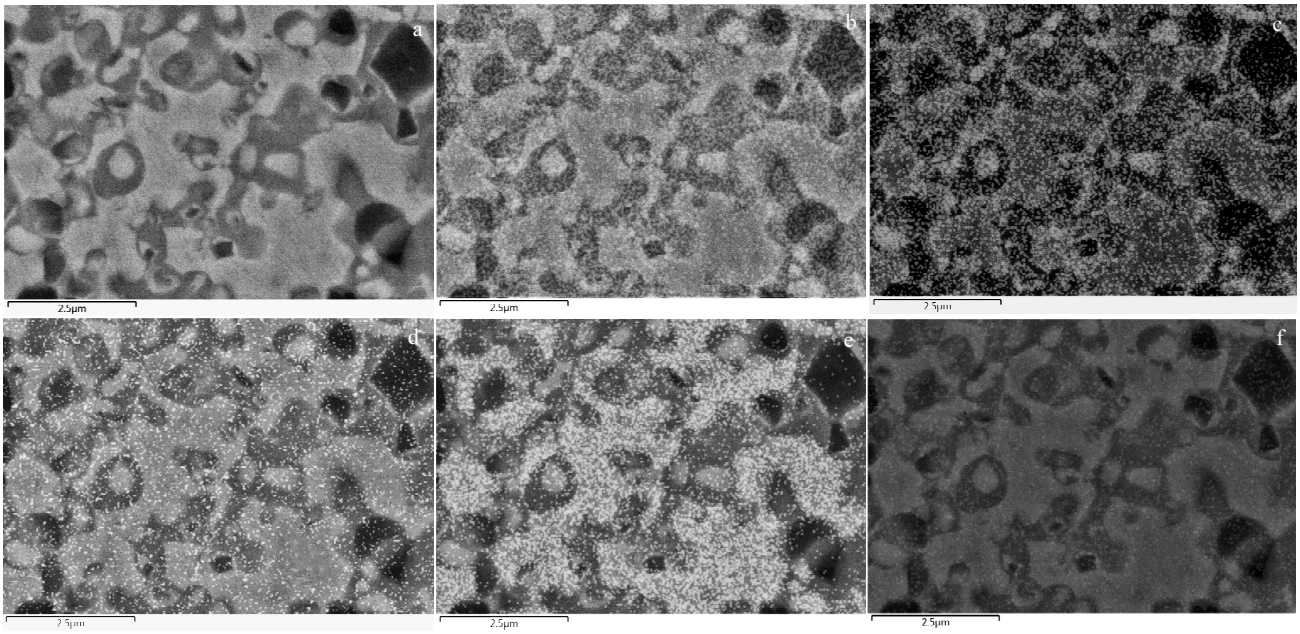

Fig.8 SEM image (a) and EDS elemental mapping of the matrix of cermet B: (b) Ti, (c) W, (d) Mo, (e) Co, and (f) C

illustrated in previous works, such as black core/grey rim, black core/white inner rim/grey outer rim, white core/grey rim and coreless structure[7,8,15-17]. A new hard phase with the black core/charcoal grey inner rim/white inner rim/grey outer rim structure, which was seldom reported, was found as shown in Fig.6c and 6d. The formation of the above new phase can be explained as follows: Firstly, the hard phase with the black core/grey rim structure on the surface of whisker-like fine granules has an opportunity to contact the WC and Co powders. Secondly, during the solid sintering stage, WC can be decomposed and diffuse through the diffusion path in Co to the surface of hard phase with black core/grey rim structures. Then the discontinuous white inner rim structure is formed on the surface of the hard phase with the black core/grey rim structure to form the black core/charcoal grey rim/white inner rim structure. Finally, the continuous grey outer rim is formed on the surface of the three layer structure, hence obtaining a new phase with the four-layer structure by dissolution-precipitation mechanism during the liquid sintering stage. The different colors of the hard phase with the four-layer structure are mainly due to the difference in chemical composition of each layer contained under SEM-BSE mode in which case heavier elements like W and Mo show a whiter color, while lighter elements like Ti,C and N show darker colors. The hard phase in the starting granules with the typical black core/grey rim structure obtained during the liquid sintering stage of cermets was widely reported, because the grey rim is rich in Mo element and shows the whiter color, described namely as the word "grey", than the black core which contained mainly Ti and C elements. During solid sintering stage, WC is almost decomposed, and part of W and C elements diffuse through the diffusion path in Co to the surface of the black core/grey rim structure of starting granules, and then form a discontinuous white inner rim around the black core/grey rim structure. At the same time, the Mo element in the grey rim mentioned above should diffuse to the black core surrounded by the grey rim, resulting in an increase in the thickness of the grey rim, which leads to a decrease in Mo element and then causes a change in color from grey to dark grey. During the liquid sintering stage, more W and C elements diffuse to the surface of third layer to form the continuous grey outer rim around the third layer by the diffusion in liquid Co, hence forming a new phase with four-layer structures. Meanwhile, the diffusion behavior of Mo element mentioned above also occurs, which further leads to the growth of rim, and then causes a change in color from dark grey to charcoal grey. A schematic diagram for describing the formation mechanisms of black core/charcoal grey inner rim/white inner rim/grey outer rim structure is presented in Fig.9.

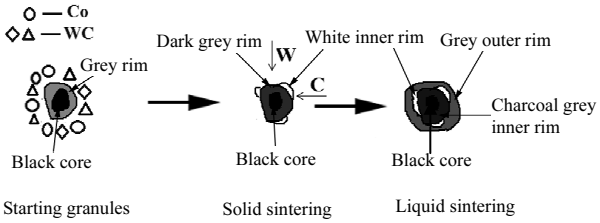

Fig.9 Schematic of the formation mechanism of black core/charcoal grey inner rim/white inner rim/grey outer rim structure

2.2 Effect of WC addition on the microstructure

It is reported that the fracture toughness of Ti(C,N) based cermets with double structure is higher than that of the conventional Ti(C,N) based cermets, while the TRS and the hardness are lower[18]. In order to preserve the TRS and hardness but not to decrease obviously, the effect of various WC additions on the microstructure and mechanical properties of the double structure Ti(C, N) based cermets were investigated. It can be clearly seen from Fig.10 that the granules are homogeneously distributed within the matrix, and have little change in the content because of the little change in volume fraction caused by changing WC. The microstructure of the matrix observed from Fig.11 indicates that the amount of white core/grey rim and coreless structures increases obviously with the increase of WC content.

In order to better understand the hard phases, the Ti(C,N) peak (422) were subjected to XRD slow-speed scanning (0.5°/min) along the back-reflection angular range ($120° < 2\theta < 125°$), and the result is shown in Fig.12. Since the ionic radius of Ti^{4+} is 6.8 nm which is smaller than 7.0 nm of W^{4+} and 7.0 nm of Mo^{4+}, the entrance of W atom dilates the lattice parameter to some extent, which makes diffraction peaks of (Ti,Mo)C or (Ti,Mo)(C,N) shift to the left[4,17]. It is denoted that the various hard phases, such as black core/grey rim, black core/white inner rim/grey outer rim, white core/grey rim and coreless structure, have the same lattice structure and different lattice parameters compared with TiC or Ti(C,N) in some extent, which makes the diffraction peaks of (Ti,Mo)C or (Ti,Mo) (C,N) shift to the left[4,17]. The W content in each structure from low to high transition is denoted in the following order: black core in the granule, black core in the matrix, grey rim around black core in the granule, grey outer rim, grey rim around white core, white inner rim, white core and coreless structure, based on the results of Fig.7 and Table 2. It is concluded that the eight peaks of (422) in Fig.12 correlate with coreless structure, white core, white inner rim, grey rim around white core, grey outer rim, grey rim around black core in the granule, black core and black core in the granule from left to right.

2.3 Mechanical properties

TRS, hardness and fracture toughness of the double structure Ti(C,N)-based cermets with different starting granule sizes are summarized in Table 3. TRS and hardness decrease with the increase of granules size, which agrees well with the Hall-Petch formula. However, the fracture toughness has

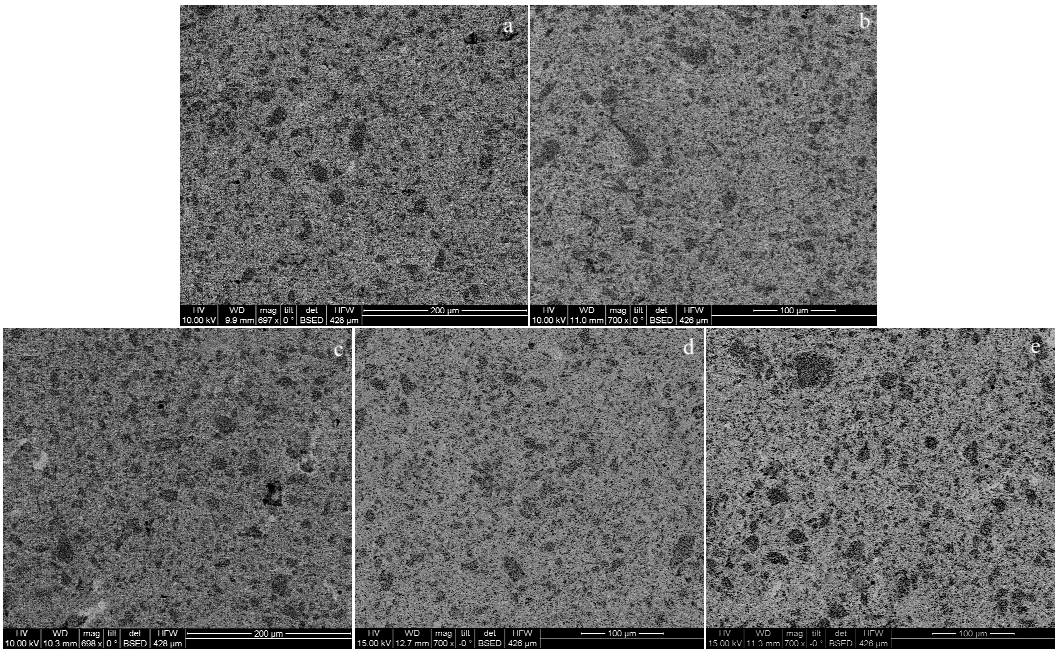

Fig.10 SEM/BSE images of granules of cermets with various WC contents: (a) 0 wt%, (b) 5 wt%, (c) 10 wt%, (d) 15 wt%, and (e) 20 wt%

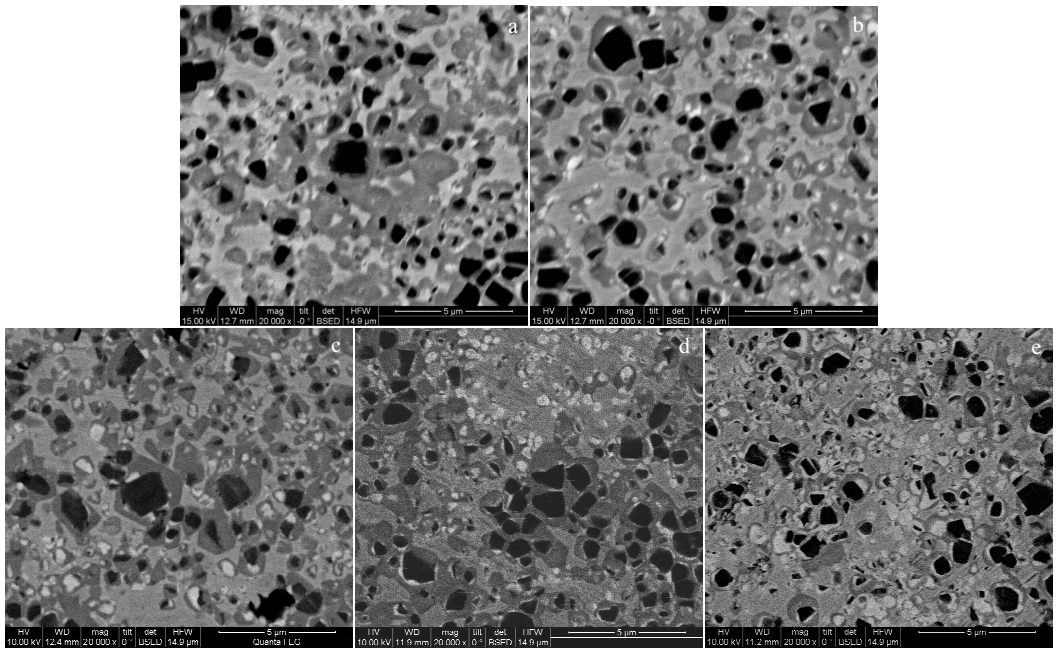

Fig.11 SEM/BSE images of the matrix of cermets with various WC contents: (a) 0 wt%, (b) 5 wt%, (c) 10 wt% , (d) 15 wt%, and (e) 20 wt%

an opposite trend. The increased fracture toughness can be mainly contributed to the higher fracture energy of coarser granules and the larger mean free path [7,10]. The effect of various WC additions on TRS, hardness and fracture toughness of the double Ti(C,N)-based cermets are plotted in Fig.13. It is denoted from Fig. 13a that the TRS increases with the increase of WC content, which can be explained by the more white core and coreless structures with higher Young's modulus due to the high content of W element. The hardness decreases with increasing WC content as shown in Fig.13b. The reason is that the lower volume fraction of granules is mainly composed of TiC. The hardness of WC and TiC is 23 and 28 GPa, respectively[19]. Therefore, the hardness decreases. With the increase of WC content, the fracture toughness increases, as

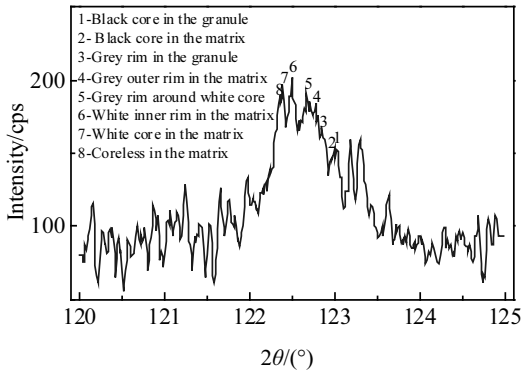

Fig.12 XRD pattern of Ti(C,N) peak (422) of the cermets with 10 wt% WC (back-reflection angular, slow speed scanning, 0.5°/min)

Table 3 Properties of double structure Ti(C,N) based cermets with different granule sizes

Sample	TRS/MPa	Hardness, HRA	K_{IC}/MPa·m$^{1/2}$
A	1057.9	88.1	11.6
B	989.1	87.7	12.8
C	895.5	87.2	13.3

shown in Fig.13c, which should be attributed to the higher Young's modulus.

2.4 Fracture morphology and behavior

The fracture morphology of the cermets with various granule sizes is shown in Fig.14, indicating that the tear ridges and pull- out effect of the granules are the main fracture modes (the effect of WC content on the fracture was omitted). The tear ridges caused by the fracture of binder in matrix, as shown by the black arrows in Fig.14, are considered as the dominant energy absorbing mechanism[20,21]. Pull-out effect of the granules, as shown by the white arrows in Fig. 14, can absorb the fracture energy to increase the crack propagation resistance, hence improving the fracture toughness. The effect of granules on the crack propagation was observed by the SEM/BSE images of the crack paths of the cermets F and A as shown in Fig. 15, using a 30 kg load by the indentation method. It can be clearly seen that crack deflection, branching, bridging and micro cracks are the main toughening mechanisms, as shown in Fig.15.

The fracture behavior of the double structure Ti(C,N) based cermet is illustrated, as shown in Fig.16, by the stress-strain curve coupled with the above toughening mechanisms. The fracture process of the cermets can be divided into three stages: elastic loading, ductile loading and crack initiation. All toughening mechanisms of this cermet including crack branching, deflection, bridging and microcracks as well as pull-out effect have a positive effect on the crack propagation after crack initiation[22-28].

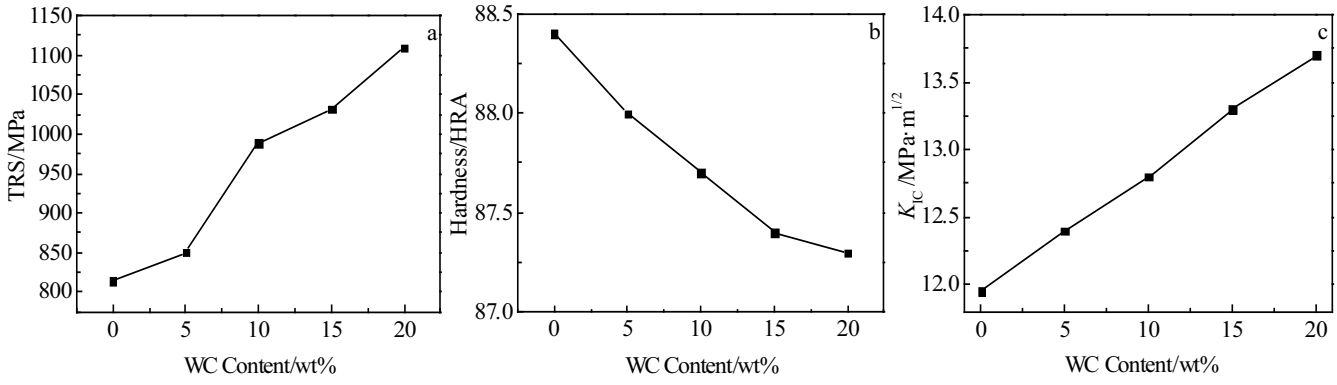

Fig.13 Effect of WC content on the transverse rupture strength (a), the hardness (b), and the fracture toughness (c)

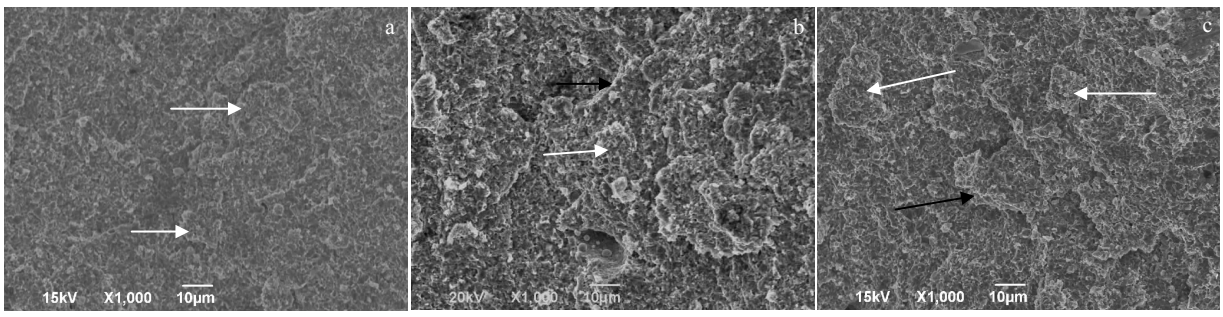

Fig.14 SEM/SE images of fracture surface of the cermet with different granule sizes: (a) 17 μm, (b) 35 μm, and (c) 48 μm

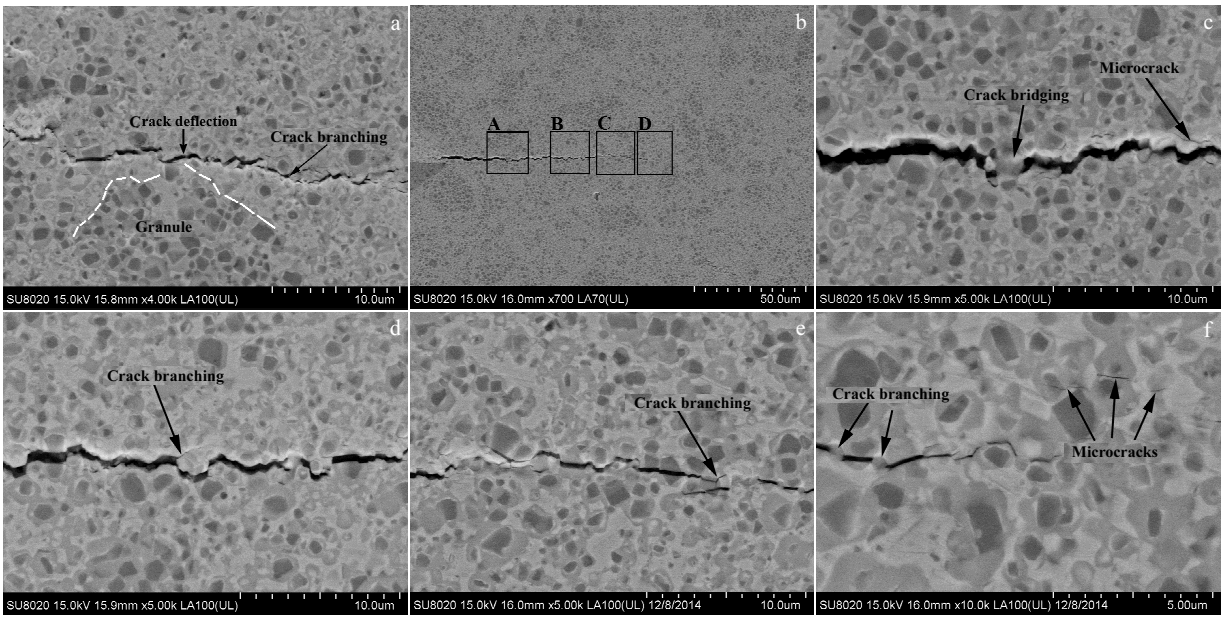

Fig.15 SEM/BSE images of the crack propagation path: (a) cermet F (30 kg load), (b) cermet A (30 kg load); higher magnification of region A (c), region B (d), region C (e), and region D (f) in Fig. 15b

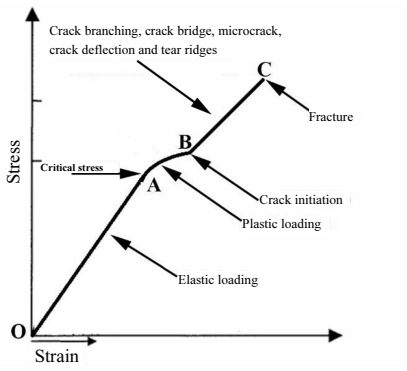

Fig.16 Stress-strain curve of double structure Ti(C,N) based cermets

3 Conclusions

1) Double structure Ti(C, N) based cermets with various granule sizes and WC contents exhibit a homogeneous distribution of the granules in the matrix. The hard phases with the white core/grey rim as well as the coreless structures increase with the increase of WC content. A new phase with the black core/charcoal grey inner rim/white inner rim/grey outer rim structure is also found.

2) With the increase of the starting granules size, fracture toughness increases obviously, while TRS and hardness decrease. With the increase of WC content, TRS and fracture toughness increase, while the hardness (HRA) shows an opposite trend.

3) The crack branching, deflection, bridging and microcracks as well as pull-out effect are the main toughening mechanisms of this cermet.

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金属陶瓷颗粒尺寸与 WC 含量对双结构 Ti(C,N)基金属陶瓷组织和性能的影响

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摘　要：研究了原始金属陶瓷颗粒尺寸和 WC 添加量对双结构 Ti(C,N)基金属陶瓷组织和性能的影响。结果表明，Ti(C,N)基金属陶瓷颗粒在基体中均匀分布。随着 WC 含量的增加，基体上的白芯/灰壳和无芯组织逐渐增多。此外，还发现了一种具有四层复合结构的新相。随着颗粒尺寸的增加，断裂韧性增加，而抗弯强度和硬度呈相反的趋势。随着 WC 含量的增加，断裂韧性和断裂强度增加，硬度降低。双结构 Ti(C,N)基金属陶瓷的主要韧化机理是裂纹分叉、桥接、偏转以及主裂纹尖端附近微裂纹的形成和颗粒的拔出效应。

关键词：双结构 Ti(C,N)基金属陶瓷；组织；性能；韧化机理

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