Effect of VC additions on the microstructure and mechanical properties of TiC-based cermets

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Abstract
VC is important carbide additive and has a similar crystal structure with TiC. In this study, the effect of adding VC amounts at content levels of 1–8 wt% on the microstructure evolution and mechanical properties of TiC-based cermets is examined. The specimens were characterized by scanning electron microscopy observations, energy-dispersive x-ray spectroscopy analysis, Image-Pro plus statistical analysis, transverse rupture strength tests, and hardness tests. The results show that VC addition is beneficial to promote the liquid-phase sintering process. When the VC addition amount is lower than 4 wt%, added VC mainly distributes in the surrounding outer rims. With increasing VC additive, the black cores are slowly dissolved and the average thickness of rims increases slightly. Many dimples exist in the fracture morphology and the strength increases gradually. Furthermore, when the VC content increased to 6–8 wt%, a part of VC is solid-dissolved with TiC, black cores with large grain appear and surround with thick outer rims. The large core-rim structure exhibits transgranular fracture mode and causes the toughness decreased.

1. Introduction
Cermet s prepared using powder metallurgy play an important role in the cutting field. Compared with the typical WC-based cemented carbides, TiC- or Ti(C,N)-based cermets have low friction coefficient and high-temperature performance, giving the cutting workpiece good surface smoothness [1–3]. Vacuum sintering has been extensively used in industrial production because it offers stable atmospheric control and large-scale production [4]. Many studies report the effects of different factors on microstructure change and properties of Ti(C,N)-based cermets. However, preparing Ti(C,N)-based cermets requires pressure sintering or adjustment of the nitrogen partial pressure to reduce porosity because of denitrifaction, thereby increasing the material’s density [5, 6]. These problems are avoided in preparing TiC-based cermets under vacuum sintering. Nevertheless, the wettability of TiC by the metal in cermets is poor and the strength of the interface is low, leading to TiC-based cermets with high brittleness and limits their industrial applications [7]. Transition metal carbides exhibit better wettability with the metal binder than TiC, which favours liquid-phase sintering. The surrounding structure formed because of the addition of transition metal carbides can effectively hinder the direct aggregation of TiC particles, improve the wettability of TiC by the metal binder and the overall mechanical properties of the sintered material [8–10]. The added transition metal carbides are then divided into two categories based on their similarity to the TiC crystal structure. For example, the crystal structures of VC, TaC, and NbC are the same as that of TiC, whereas the crystal structures of Mo2C, WC, and Cr3C2 differ from that of TiC. The carbides whose crystal structure differs from that of TiC exhibit great solubility in the metal binder, among which Mo2C and WC exhibit higher solubility in the binder [11, 12]. Note that Cr3C2 is enriched in the binder and at the phase boundary, which can suppress crack propagation [13]. The carbides with the same crystal structure as TiC have lower solubility in the binder and easily form (Ti, M) C solid solutions, where M denotes the solid dissolved V, Ta, Nb atoms. Because Ta and Nb have considerably larger atomic radii than Ti,
the unit cell distortion of (Ti, M) C (M = Ta, Nb) increases and affects the dissolution of carbides in the binder [14]. Compared with other transition-metal carbides, VC is special. VC and TiC have the same NaCl-type face-centred-cubic crystal structure and form continuous substitutional solid solution. Hence, a systematic study of the effect of VC additive on the structural evolution and mechanical properties of TiC-based cermets would be meaningful. As a trace additive (≤1 wt%), VC has been used as an inhibitor for grain growth in WC-based cemented carbides with good results. This grain growth inhibition is enriched at the phase boundary and the coarsening of carbide grains are effectively suppressed during liquid-phase sintering [15]. Huang et al suggested that NbC has the same crystal structure as TiC/TiCN, indicating that research related to TiCN-based cermets is applicable to NbC-based cermets [16–18]. In the NbC-12 vol% Ni system studied by the author, the addition of 5–10 vol% of VC can improve the wettability of the carbides and binder of NbC-based cermets and VC can improve toughness compared to Mo2C [19].

Investigation of VC addition on the microstructure and mechanical properties of TiC- or Ti(C,N)-based cermets has less been reported. Commonly, VC is considered as the grain growth inhibitors with small amounts. Feng et al and Zhan et al studied the effect of VC addition on grain growth inhibition of Ti(C,N)-based cermets with less than 1 wt% and the grain growth was effectively prevented [20, 21]. Zhang et al attempted to add VC with the amount of 0–1.5 wt% as grain growth inhibitors [22]. But the authors found that the mechanical properties decreased with increasing VC content. To exert the solution strength effect, Arenas et al studied the effect of higher VC contents with more than 18 wt% on the microstructure and properties of TiC-based cermets [23]. They found that higher quantities of VC led to the surrounding rims too thick and the mechanical properties sharply decreased. It is known that core-rim structure forms via the dissolution and precipitation process. Lattice misfit at the core-rim interfaces leads to the formation of internal stress and decreased the mechanical properties. To reduce the difference of lattice parameters between the core-rim structure, Xiong et al recently added VC within 0–6.4 wt% and found that the mechanical properties were obviously improved with VC addition of 4.8–6.4 wt% [24]. It seems like that VC with the content of lower than 10 wt% could exert the role of strengthening core-rim structure. However, no papers were found to report the effect of VC on the microstructure and properties of TiC-based cermets within this range. Interestingly, we found that the addition of VC content was closely related to the designed composition and the microstructure of TiC-based cermets was improved with proper VC content. To compare with the small amount of VC as grain inhibitor, in this study, high VC content within 1–8 wt% on the microstructure and mechanical properties of TiC-based cermets was systematically studied under a vacuum-sintering atmosphere, thereby providing a theoretical basis for expanding the application of VC in TiC-based cermets.

2. Experimental

2.1. Preparation of TiC-based cermets

TiC-based cermets were prepared by commercially available TiC, VC, WC, Mo2C, Co, and Ni micron powders. The purity of the raw material powder is 99 wt%, and the average particle size is <3 μm. Commercial powders were mixed in the mass proportions of TiC - xVC — 3 wt%WC — 3 wt% Mo2C — 1.9 wt%Co — 7.8 wt%Ni. The powder mixtures with 0 wt%, 1 wt%, 2 wt%, 4 wt%, 6 wt%, 8 wt% VC are named cermet A, cermet B, cermet C, cermet D, cermet E and cermet F, respectively. Mixed powders were wet-milled at a rotation speed of 200 rpm in a planetary ball mill (YXQ-4L) with alcohol as the ball-milling medium and the ball-to-material ratio was 10:1. The grinding balls were WC–Co sintered carbide balls. The ball-milling time was 72 h with a ball-milling interval of 10 min per 2 h. After drying in vacuum at 60 °C, a SD-2X rubber—gasoline solution was then used as a forming agent. Under a pressure of 200 MPa, the mixtures were unidirectionally pressed into green compacts of 25 mm × 8 mm × 8 mm using a hydraulic press. TiC-based cermets were then prepared by vacuum sintering of the green compacts at a sintering temperature at 1420 °C and for a holding time of 1 h.

2.2. Properties tests, phase composition and microstructure tests

The microstructure evolution and fracture surface morphology of the TiC-based cermets were observed using a TESCAN VEGA II LMU scanning electron microscope (SEM) in backscattered electron (BSE) mode and second electron (SE) mode, respectively. The grain size was measured using an image analysis method [25]. Elemental composition of cores, rims and binders was analysed using the built-in energy-dispersive x-ray spectrometer (EDS) of the electron microscope. The phase identification was performed on a DX-2700-type x-ray diffractometer equipped with a Cu radiation source (wavelength: 1.540 56 Å) operated at a tube voltage of 40 kV and the scanning step length was 0.02°.

The hardness was tested on a THV-50DX Vickers hardness tester using a load of 30 kgf and a holding time of 15 s. The fracture toughness (KIC) was calculated according to the indentation method proposed by Shetty et al [26]. Transverse rupture strength (TRS) was measured using a three-point bending method performed on a
WDW-50A universal mechanical testing machine. Five samples were then tested and averaged with a test span of 17 mm and a loading speed of 0.5 mm min$^{-1}$.

3. Results and discussion

With increasing VC content, the BSE microstructure of the TiC-based cermet varies (figure 1). The microstructure is primarily a typical black core-grey rim structure uniformly distributed in a white binder, and a small amount of white core-grey rim structure is observed in which the white core is fine submicron particles. According to the dissolution–precipitation theory of the formation of core-rim structures, the solubility of TiC in Co and Ni is limited, when the binder content is small, undissolved TiC particles form black cores$^{[27, 28]}$. During the solid-state sintering stage, Mo, W, and V atoms in the additive diffuse to the core of fine TiC particles to form a heavy-metal-enriched carbide solid solution (Ti,Me$^+$$)_C$, where Me is dissolved Mo, W, and V atoms. Due to higher average atomic number, it is observed as white cores under backscattering electronic contrast
(green arrow in figure 1(a)) [29]. However, when Mo, W, and V atoms diffuse to the surface of large-particle TiC, because of the slow diffusion rate of atoms in the solid-state sintering stage, the (Ti,Me)C solid solution surrounds the black cores to form a thin inner rim (red arrows in figure 1(a)) [30]. During the liquid-phase sintering stage, metal atoms dissolved in the binder exhibit fast diffusion. A considerable amount of (Ti,Me)C solid solution precipitates and forms thick outer rims. Because the heavy-metal elements in the outer rims are present in lower concentrations than in the inner rims and white cores, and because their average atomic number is greater than that of TiC, the outer rim appears grey under the backscattering electronic contrast and the inner rim is light grey. The contrast between the inner rim and outer rim is more distinct in figure 1(g). The core–rim structure jointly constitutes the hard phase of TiC-based cermet and contributes to hardness.

When VC additive is 0 wt%, the boundary of inner rims in figure 1(a) is clear and the inner rims develop well. When VC is added, the rims are primarily composed of grey outer rims and the thickness of the inner rims decreases. Comparison indicates that more inner rims are dissolved by liquid binder with VC additive. It is noted that the core–rim structure exhibit a substantial change with VC content higher than 6 wt%. A part of black cores with bigger grain size and thicker rims appear.

To investigate the effect of VC additives on the microstructure evolution, the grain size distribution, average grain size of the black cores and average thickness of the rims are analyzed and the statistical results are shown in figures 2 and 3. Under small VC addition amounts (lower than 4 wt%), black cores with a grain size of lower than 1.0 μm account for a larger proportion. Due to preferential dissolution of small TiC grains, main changes in the grain size distribution of black cores focus on the area less than 1.0 μm. Hence, the average grain size of the black cores decreased with a small fluctuation. The changes reflect the dissolution rate of carbides and finally affect the thickness of outer rims. The more carbides dissolved in the binder, the more (Ti,Me)C solid solution precipitated. Thus, the thickness of the grey outer rims increases slowly with a concomitant decrease in the average grain size of the black cores. Comparison of the curves suggests that the addition of a small amount of VC promotes the liquid-phase sintering process. This result agrees with Xiong’s and Lin’s work, which pointed that VC accelerated the dissolution of carbides in the binder of TiC-based cerments [24, 31]. When VC addition amount is higher than 6 wt%, the proportion of black cores with a grain size of larger than 2.0 μm is increased, causing a sudden increase in the average grain size of black cores. The formed large black cores are surrounded by thicker rims and thus the average thickness of the grey outer rims increases.

The observed microstructure evolution of core–rim structure may be explained as follows: WC and Mo2C have higher solubility in the binder. The 3 wt% WC and 3 wt% Mo2C additives are dissolved in the liquid-sintering stage. VC additive is beneficial to promote the dissolution of TiC in binder and improves the liquid-sintering process. When the VC additive is lower than 4 wt%, the promotion effect is slow, causing the average grain size of black cores decreased slightly and the average thickness of grey outer rims increased slowly. However, when the VC content is higher than 6 wt%, besides the dissolution of added VC in the binder, a part of VC are solid dissolved with TiC to form (Ti,V)C solid solution. Because smaller TiC particles are easier to dissolve in the binder, the VC combined with large grain TiC and lead to the uneven distribution of core–rim structures shown in cermet E and cermet F. Moreover, thick rims surrounding the large particle cores indicate that the amount of carbides dissolved in the binder is increased compared to the cerments with lower VC content.

To understand element distribution in the cores, rims, and binder phase, the EDS results of TiC-based cerments with different VC contents are shown in table 1. Because the thickness of inner rims in TiC-based cerments with added VC is small, the EDS point-scanning results of the inner rims are considerably affected by the adjacent black cores and the grey outer rims. Thus element distribution is analyzed in the outer rims. Comparison of the distribution of metal elements in different phases reveals that Mo, W, and V are primarily distributed in the outer rims, then in the binder, with the lowest content in the cores. These results indicate that the primary functions of the Mo2C, WC, and VC additives are to dissolve in the binder during the liquid-phase sintering stage and then precipitate during the cooling stage to form the outer rims. The increase in VC content promotes the liquid-phase sintering process, resulting in progressively increasing Mo, W, and V contents in the binder. When small amounts of VC with lower than 4 wt% are added, V exhibits an extremely low content in the black cores and is primarily distributed in the outer rims. When the VC addition amount is increased to 6–8 wt%, the V content in the detected large-particle black cores substantially increases, indicating that some of the VC particles are solid-dissolved with TiC to form a (Ti,V)C solid solution, which is consistent with the above explanation.

TiC functions as a hard-phase matrix, and Ti exhibits the highest content in the cores and rims. The Ti content in the cores exhibits small fluctuations overall as the VC content increases. This behavior is explained as follows. To achieve more accurate composition results of the EDS point scanning, we performed spot analysis at the centre of the large-particle black cores. Because the large-particle black cores are mainly undissolved TiC, the composition change in the central part was hardly influenced by the adjacent areas and varied VC content. With increase in VC addition amount, the Ti content in the rims exhibits small fluctuations, showing a slow increasing trend overall. According to the previous analysis of the grain size distribution of the black cores, the addition of
VC in amounts lower than 4 wt% promotes the dissolution of TiC with a slow speed, resulting in a slow increase in the Ti content in the precipitation rims.

XRD lines of TiC-based cermets with varied VC additive are shown in figure 4. The diffraction lines of TiC-based cermets only contain two kinds of phases with cubic crystal structure, that is, the ceramic phase and the binder. It is seen that the diffraction lines of Mo₂C, WC, and VC additives disappeared and the additives existed in the form of (Ti,Me)C solid solution. This change indicates that the added Mo₂C, WC, and VC mainly contribute to ceramic phase. There is little difference in the lattice parameters between cores and rims, the diffraction lines are not separated from each other. With increasing VC content, the (200) diffraction lines of (Ti,Me)C firstly shift to lower angles and then shift to higher angles gradually. Reasons for this change may be explained as follows: The atomic radii of V (Rᵥ = 0.1338 nm), Mo (Rₘ₀ = 0.1386 nm) and W (Rₚ = 0.1394 nm) are smaller than that of Ti (Rₜᵢ = 0.1467 nm). VC additive is beneficial to promote liquid sintering. Compared with Cermet A without VC additive, more carbides precipitate from the binder in Cermet B and the lattice parameters of (Ti,Me)C increase. However, with increasing VC content, the lattice parameters of (Ti,Me)C decrease due to the increased V content in outer rims with smaller atomic radius. The unit cell distortion of the binder increases due to the dissolved Ti, W and
Figure 3. Average grain size and thickness of rims in TiC-based cermets with different VC contents. (a) relationship between average grain size and VC content, (b) relationship between average thickness of rims and VC content.

Figure 4. XRD diffraction lines of TiC-based cermets with different VC contents.

| Table 1. Chemical composition of black cores, grey rims and binder in TiC-based cermets measured by EDS. |
|---------------------------------|-----|-----|-----|-----|-----|-----|-----|
| Element (at.%)                  | Ti  | V   | Mo  | W   | Co  | Ni  | C   |
| Black core                      |     |     |     |     |     |     |     |
| Cermet A                        | 70.85 | 0 | 0.55 | 0.81 | 0.18 | 0.6 | 27  |
| Cermet B                        | 70.51 | 0.13 | 0.57 | 1.23 | 0.16 | 0.46 | 26.94 |
| Cermet C                        | 70.52 | 0.26 | 0.46 | 0.95 | 0.08 | 0.31 | 27.41 |
| Cermet D                        | 70.35 | 0.32 | 0.15 | 0.41 | 0.03 | 0.10 | 28.64 |
| Cermet E                        | 70.13 | 1.11 | 0.28 | 0.57 | 0.09 | 0.29 | 27.53 |
| Cermet F                        | 70.04 | 1.42 | 0.32 | 0.87 | 0.07 | 0.08 | 27.20 |
| Grey rim                        |     |     |     |     |     |     |     |
| Cermet A                        | 58.2 | 0   | 1.23 | 2.01 | 1.73 | 4.61 | 32.22 |
| Cermet B                        | 60.11 | 0.51 | 1.38 | 2.48 | 1.28 | 3.05 | 31.19 |
| Cermet C                        | 60.17 | 1.41 | 1.41 | 2.89 | 0.14 | 0.14 | 33.65 |
| Cermet D                        | 63.67 | 2.8 | 1.41 | 3.16 | 0.18 | 0.25 | 28.53 |
| Cermet E                        | 63.87 | 4.68 | 1.49 | 2.57 | 0.53 | 1.42 | 25.44 |
| Cermet F                        | 64.85 | 4.64 | 1.1 | 2.23 | 0.08 | 0.28 | 26.81 |
| Binder phase                    |     |     |     |     |     |     |     |
| Cermet A                        | 31.52 | 0   | 0.59 | 1.08 | 8.98 | 27.61 | 30.23 |
| Cermet B                        | 23.27 | 0.28 | 0.64 | 1.29 | 11.51 | 38.3 | 24.71 |
| Cermet C                        | 27.33 | 0.56 | 0.65 | 1.34 | 12.82 | 41.76 | 15.55 |
| Cermet D                        | 20.16 | 1.59 | 0.64 | 1.44 | 14.07 | 47.6 | 14.51 |
| Cermet E                        | 20.21 | 2.2 | 0.86 | 1.68 | 12.27 | 38.58 | 24.21 |
| Cermet F                        | 39.28 | 4.08 | 0.92 | 2.23 | 7.33 | 22.98 | 23.19 |
Mo with large atomic radius than that of Co and Ni. Therefore, the (111) diffraction lines of binder shift to lower angles with increasing VC additive.

Figure 5 shows the effect of the VC addition amount on the mechanical properties of TiC-based cermets. With increasing VC addition amount of lower than 4 wt%, the TRS and hardness increase gradually. The increase in VC content promotes liquid-phase sintering, increasing the wettability of the ceramic carbides and binder, thereby improving the TRS of the material. More carbide dissolve in the binder and the hardness is increased due to solution strength effect. However, when the VC addition amount is higher than 6 wt%, the TRS no longer increases and hardness decreases. Reasons can be explained as follows: the appeared large-particle cores with thick rims in cermet E and cermet F increase the material brittleness, which hinders the improvement in TRS. Furthermore, the hardness of surrounding rims is lower than that of black cores, leading to the measured hardness decreased [32, 33]. When VC additive is 4 wt%, cermet D owns the best mechanical properties with a bending strength of 1028 Mpa and a Vickers hardness of 1640 N mm\(^{-2}\).

To further understand the influence of VC additive on the \(K_{IC}\) of TiC-based cermets, the fracture surface morphology is observed and shown in figure 6. When the VC additive is lower than 4 wt%, dimples are typical characteristics in the fracture morphology. The dimples are formed because the ceramic particles are pulled out during the process of fracture [34–36]. This kind of fracture mode need to consume large amounts of energy and is beneficial to increase the \(K_{IC}\). The \(K_{IC}\) of TiC-based cermets fluctuated first within a small range and then increased to a peak value. However, when the VC additive is higher than 6 wt%, breaking sections of core-rim structure with large particles are observed (marked with red squares). The appearance of these smooth breaking sections is due to the formed large particle black cores with thick rims in BSE microstructure of cermet E and cermet F. This kind of fracture mode belongs to the transgranular fracture and is harmful to the \(K_{IC}\). Thus, under higher VC content, the \(K_{IC}\) of cermet E and cermet F is decreased.

4. Conclusion

(1) VC addition promotes the liquid-phase sintering process and more carbides dissolved in the binder. When the VC addition amount is lower than 4 wt%, added V primarily distributed in the outer rims and the contents of metal elements in the binder gradually increase. The average particle size of black cores decreases slowly with a concomitant slight increase in outer rims. TRS progressively increases to the peak value.
When the VC content increases to higher than 6 wt%, a part of VC are solid dissolved with TiC to form 
(Ti,V)C solid solution, resulting in a substantial increase in the V content in black cores. The appeared large 
particle black cores are surrounded with thick rims, causing the average particle size of black cores and 
thickness of rims increased. The TRS no longer increases and KIC decreases.

Dimples are typical characteristics in the fracture morphology of TiC-based cermets with VC additive lower 
than 4 wt%. Continued to increase VC content, smooth breaking sections of large particle core-rim 
structure with transgranular fracture mode exist and decreased the material toughness. When the VC 
addition amount is 4 wt%, TiC-based cermet exhibits optimal mechanical properties.
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