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Effect of WC content on the microstructures and corrosion behavior of Ti(C, N)-based cermets

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Abstract

The influence of WC content on the microstructure and corrosion behavior of Ti(C, N)-based cermets in 2 mol/L nitric acid solution was studied in this paper. There exists typical core/rim structure in the cermets. The cores appear black or white, and the rim is divided into white inner rim and grey outer rim. The undissolved Ti(C, N) particles normally appear as black cores, while the white core, inner rim and outer rim are (Ti, W, Mo) (C, N) solid solution formed at different sintering stages. The inner rim and white core appear brighter atomic contrast than the outer rim and black core, which is attributed to their higher W and Mo content. The thickness of the inner rim increases with WC addition, but the grain size of core/rim phase becomes finer. Meanwhile, the amount of white cores increases and that of black cores decreases. WC is more easily oxidized and dissolved in the nitric acid solution, compared with Ti(C, N). Therefore, the degradation of inner rim phase and the white core becomes more considerable with the increase of WC content. Consequently, the corrosion rate of cermets increases and the corrosion resistance of Ti(C, N)-based cermets is deteriorated with the increase of WC content.

Keywords: B. Microstructure; C. Corrosion; D. Ti(C, N)-based cermets

1. Introduction

Ti(C, N)-based cermets have high hardness, enhanced strength, oxidation resistance at high temperature, perfect thermal stability, low friction coefficient to metals and outstanding wear resistance [1–3]. Ti(C, N)-based cermets, therefore, are widely applied in many fields, such as mechanical processing, transportation, petrochemical engineering and so on [4]. In these industrial applications, large quantities of components are increasingly manufactured with cermets, such as cutting tools, seal rings, pumps, choke valves, jet nozzles, saw blades and fluid mixers [5–7]. These components, however, not only are subjected to severe wear, but also contact directly with corrosive liquid and suffer from terrible corrosion in the chemically aggressive environments [2,7–11]. Therefore, both enhanced wear resistance and excellent corrosion resistance are required for a long lifetime and reduction of cost [12]. Because cermets have high wear resistance owing to the hard phase [7], the research on the corrosion behavior of cermets has become an inevitable trend.

At present, the corrosion resistance of Ti(C, N)-based cermets was reported in a very limited extent. Qian and Xiong [13] pointed out that the corrosion resistance of Ti(C, N)-based cermets was controlled by binder′s corrosion in the solution of 5% HNO_3 and 50% NaOH. The corrosion resistance of cermets increased with the decrease of Ni binder phase. Cermets corrosion behavior was galvano-chemistry corrosion protected by passivation film. Li et al. [14] concluded that the Ti(C, N)-based cermets for surgical cutting tools had good corrosion resistance and the corrosion behavior of the tested cermets was mainly of galvano-chemistry in the solution of hypochlorite, but of oxidation in the solution of H_2O_2. The electrochemical behavior of Ni, Ti(C, N) − 20Ni, and Ti(C, N) − 20Ni − X

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(X: WC/NbC/TaC/HfC) was documented in the literature [15] by potentiodynamic polarization tests in freely aerated 0.2 mol/L H_2SO_4 solution. Manoj Kumar found all the materials (except HfC-containing cermet) exhibited active–passive polarization behavior, characterized by two distinguishable passive regions. It was reported that the corrosion of hardmetals in neutral and acidic media usually resulted in a preferential dissolution of the binder phase leaving a skeleton of carbides [16,17].

From the above, there are already some reports about the corrosion behavior of Ti(C, N)-based cermets. But it is unknown that whether WC can improve the corrosion resistance of cermets or not. In Ti(C, N)-based cermets, WC is also a kind of an essential ingredient, similar to Mo_2C [18,19]. According to relevant reports in the literatures [20,21], up to now, the survey on cermets was focused on the effect of WC content on the microstructure and mechanical properties of Ti(C, N)-based cermets. WC could improve the wettability of Ni binder phase to the hard phase Ti(C, N), leading to better densification. It was found that the microstructure of ultrafine Ti(C, N)-based cermets became finer with the increase of WC addition [1,22]. Results also showed that the transverse rupture strength and the hardness increased as W element increased in the materials. In a word, WC plays an important role in the mechanical properties of the cermets.

Based on the published literatures, there is limited information about the corrosion behavior of Ti(C, N) -based cermets and the effect of WC addition on the microstructure and mechanical properties of the material; however, it has not been explored systematically about the corrosion properties of Ti(C, N) − Ni cermets with various WC content in an acidic environment. Hence, in the present work, $Ti(C,\ N)-15Ni-10Mo_2C-x$WC cermets are prepared by conventional powder metallurgical technique, and the corrosion behavior of Ti(C, N)–15 wt% Ni cermets with different WC content is studied in 2 mol/L HNO_3 solution. Furthermore, the corrosion mechanism is also explored and analyzed in detail.

2. Experimental procedures

2.1. Specimen preparation

The characteristics of raw powders commercially available in this work are given in Table 1. After weighing, various mixtures were respectively blended with gasoline in a tumbling ball mill for 72 h at a ball-to-powder weight ratio of 10:1. WC–8 wt% Co balls with a diameter of 10 mm were used as milling bodies and the milling speed was 68 r/min. After milling the slurry powders were dried and then mixed with rubber as the pressing aid. At last, green compacts of dimensions 20 mm × 6.5 mm × 5.25 mm were pressed under uniaxial load of 100 MPa and were subsequently sintered at 1440 °C for 1 h with the vacuum degree of 5 Pa. All samples were prepared by the same method, and their nominal compositions are listed in Table 2. The sintered samples were ground to remove the surface oxide layer and obtain parallel faces and then polished to a 1 μm diamond finish with diamond paste to observe the microstructure. The microstructures of samples were observed by scanning electron microscopy (SEM). The actual densities of cermets were measured by the Archimedes method. The relative densities of cermets are the ratio of actual densities with theoretical densities.

2.2. Immersion test

Five samples of each grade cermet were obtained from the polished samples and the size of each specimen was measured by a micrometer. The samples were cleaned by ultrasonic with acetone and alcohol and then dehydrated at 80 °C for 24 h. After completing these steps, each sample was weighed using the precision electron balance model BSA124S (Beijing Sarturius Co., Ltd., China) with 0.1 mg precision and immersed into 2 mol/L HNO_3 solution at room temperature. After every 6 h interval, the corroded samples were taken out to clean with acetone using an ultrasonic bath for 5 min in order to remove residual corrosive solution, and dehydrated at 80 °C for 24 h again. The weight of specimens was measured again for specific mass loss. Repeat this process until the total immersion time reached 48 h.

The corrosion rate was calculated using the following equation:

$$c = \Delta m / At \tag{1}$$

Table 2
Chemical compositions of Ti(C,N)-based cermets (wt%).

Cermets	$TiC_{0.7}N_{0.3}$	Ni	WC	Mo_2C
C0	Balance	15	–	10
C1	Balance	15	5	10
C2	Balance	15	10	10
C3	Balance	15	15	10

Table 1
Characteristics of raw powders.

Powders	Total carbon (wt%)	Free carbon (wt%)	Oxygen (wt%)	FSSS (μm)	Manufacturer
$TiC_{0.7}N_{0.3}$	13.52	0.08	0.30	1.85	Changsha Wing Hing High-Tech New Materials Co., Ltd., China
WC	6.19	0.07	0.29	0.45	Changsha Wing Hing High-Tech New Materials Co., Ltd., China
Mo_2C	6.03	0.20	0.52	1.55	Changsha Wing Hing High-Tech New Materials Co., Ltd., China
Ni	0.10	–	0.10	2.65	Chengdu Nuclear 857 New Materials Co., Ltd., China

where c is the corrosion rate (g m^{-2} h^{-1}), Δm is the weight loss (g), t is the immersion time (h), and A is the total surface area of sample (m^2). The final corrosion rate was determined by the average results of five samples.

In order to explore the corrosion behavior of cermets, each grade sample was immersed in 2 mol/L HNO_3 solution for 10 h and the corroded surface morphology was observed in the secondary electron (SE) mode.

3. Results and discussion

3.1. Microstructures of Ti(C, N)-based cermets

Microstructure of Ti(C, N)-based cermets before corrosion is characterized by SEM in BSE mode as shown in Fig. 1. Typical core/rim structure of conventional cermets is observed from the photograph, which has two kinds of forms. One is black core-grey rim structure, the other is white core-grey rim structure. The black core is the undissolved Ti(C, N) particles, while the white core and the rim phase are (W, Mo, Ti) (C, N) solid solution. Moreover, the rim has two layers including the inner rim and outer rim according to their relative location around the core. Although the rim phase is (W, Mo, Ti) (C, N) solid solution, the inner rim phase contains higher Mo and W than the outer rim phase. The core/rim structure is widely presented in the Ti(C, N)-based cermets as a result of the dissolution–precipitation process [20–22]. During the sintering, WC, Mo_2C and the fine Ti(C, N) particles are dissolved in Ni binder phase with the increase of temperature. During solid state sintering [18–20], owing to high dissolution rates, WC and Mo_2C are dissoluted and diffused in the binder phase rapidly. When WC and Mo_2C get saturated in the binder phase, they will produce nucleation and precipitate on the surface of the undissolved Ti(C, N) black cores. Therefore, the inner rim is formed, which is (W, Mo, Ti) (C, N) solid solution, rich in W, Mo but poor in Ti. When the black core dissolves completely, the white core is produced during the solid state sintering, similar to the inner rim phase. So the white core is also (W, Mo, Ti) (C, N) solid solution. During liquid phase sintering, the dissolved WC and Mo_2C sequentially precipitate on the surface of inner rim phase and the outer rim phase is formed, which is rich in Ti but poor in W and Mo [18–20,23–24]. Because the micrographs of the elements with larger atomic number show brighter color, both the inner rim phase and the white core show brighter than the grey outer rim phase.

Fig. 1 reveals that the thickness of rim phase increases with WC addition, while the size of the black core decreases. It is explained that more WC precipitates on the surface of black Ti(C, N) core with the increase of WC addition, resulting in the thicker rim phase. Based on the overview of the microstructures shown in Fig. 1, the grain size of core/rim structure becomes finer with the increase of WC content. Additionally, the amount of white cores increases and that of black cores decreases with the increase of WC content. It is observed from Fig. 1 that some outer rim phase disappears at last and the thickness of the inner rim phase increases, particularly in Fig. 1(d). Meanwhile, in Fig. 1(a), it is apparent that the grain size of cermets without WC addition is homogeneous. However, when the WC addition increases gradually, the

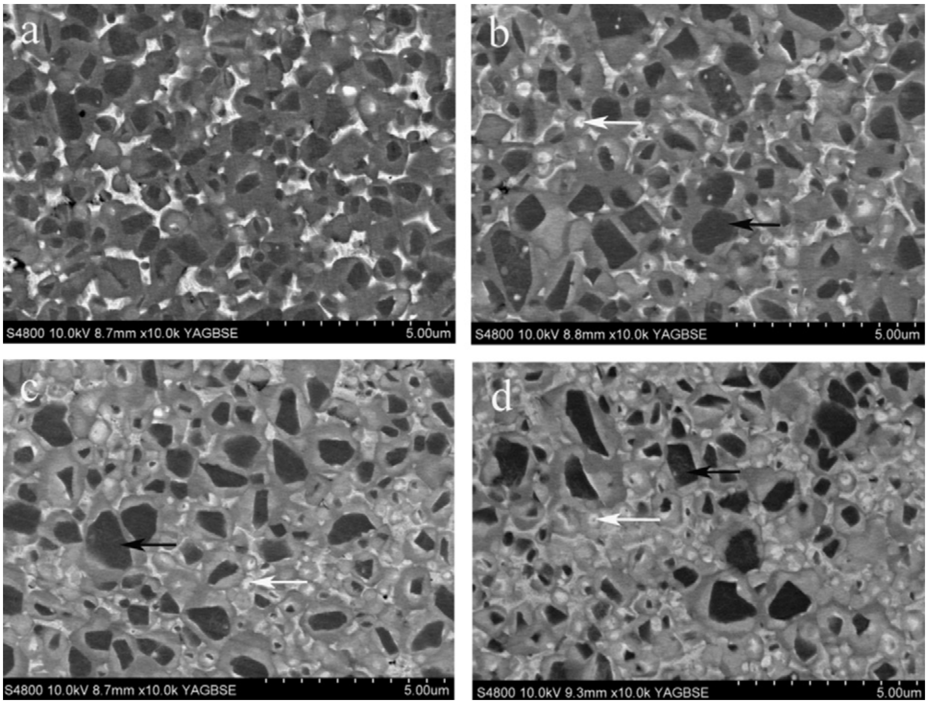

Fig. 1. SEM–BSE micrographs of Ti(C, N)-based cermets with different WC addition: (a) C0: 0 wt% WC; (b) C1: 5 wt% WC; (c) C2: 10 wt% WC and (d) C3: 15 wt% WC.

inhomogeneous cores (fine white cores shown white arrows and coarse black cores shown black arrows) are observed remarkably in Fig. 1(b), (c) and (d). During the sintering process, the finer hard phase particles is preferentially dissolved in the binder phase [3,22,25]. It is reported that WC has higher solubility than TiC/TiN in Ni binder phase [26]. Therefore, the solubility of Ti(C, N) is reduced when WC addition increases in the raw materials. The fine white core phase is formed via the dissolution and the precipitation process. However, small quantities of black core particles remain coarse because some coarse Ti(C, N) particles are not completely dissolved in the binder phase. Hence, the grain size appears inhomogeneous with the increase of WC content.

In Ti(C, N)-based cermets system, the wettability of Ni binder phase on Ti(C, N) hard phase is related to the stability of carbides. Owing to the lower negative heat enthalpy of formation of WC, as WC content increases in the raw materials, the wetting angle between the liquid binder and the carbide is smaller [27], and therefore, the wettability of Ni binder on the Ti(C, N) becomes better. Although the densification of cermets is high during fabrication, it can be further improved due to the better wettability of Ni binder phase on Ti(C, N) hard phase by adding WC. The porosities and the relative densities of cermets are listed in Table 3.

3.2. Corrosion rates

Fig. 2 shows the corrosion rates of Ti(C, N)-based cermets with different WC addition immersed in 2 mol/L HNO_3 solution for various immersion times. It is found that the trend of corrosion rate is basically consistent with the increase of the immersion time. With longer immersion time, the corrosion rate gradually reduces and at last tends to stabilize except cermet C3. Because in the beginning, the surface of the samples is clean and the exposed area in the corrosive medium is large leading to enhanced corrosion process of cermets. When the corrosion reactions occur continuously, the corrosion products cover around the surface of samples and reduce the direct contact area between cermets and aggressive medium. Hence, the corrosion products have some degree of protective effect and consequently the corrosion rate reduces. Furthermore, the corrosion rate of cermets increases with WC addition in the same corrosive environment. Nevertheless, it is observed that the corrosion rate of cermets increases regularly as WC addition increases from 0 wt% to 10 wt%, while the corrosion rate of cermet C3 increases sharply compared to cermet C2.

3.3. Corrosion morphologies and corrosion mechanism

Fig. 3 shows the corrosion morphologies of Ti(C, N)-based cermets with different WC content immersed in 2 mol/L HNO_3 solution for 10 h. The binder phase in the four groups is corroded at the attacked surface. It is observed that the remaining hard phase particles support each other and form a continuous hard phase skeleton from Fig. 3(a). But some unsupported hard phase particles fall off from the skeleton due to the degradation of binder phase, and corrosion pits appear on the hard phase skeleton evenly. It is obvious that these hard phase particles are core/rim phase, which are corroded severely, especially the inner rim phase. Therefore, the corrosion groove is found due to the degradation of the inner rim phase between the outer rim phase and the black core. It can be seen from Fig. 3(b) that the thickness of rim phase (W, Mo, Ti) (C, N) solid solution increases with 5 wt% WC addition, which is inferiorly resistant to corrosion in the nitric acid solution. This correlates well with the phenomenon that the corrosion of rim phase in cermet C1 becomes more intensive compared to cermet C0, as shown in Fig. 3(b). It is found that the continuous hard phase skeleton is damaged and a large area of hard phase particles fall off, as shown in Fig. 3(c) and (d). The hard

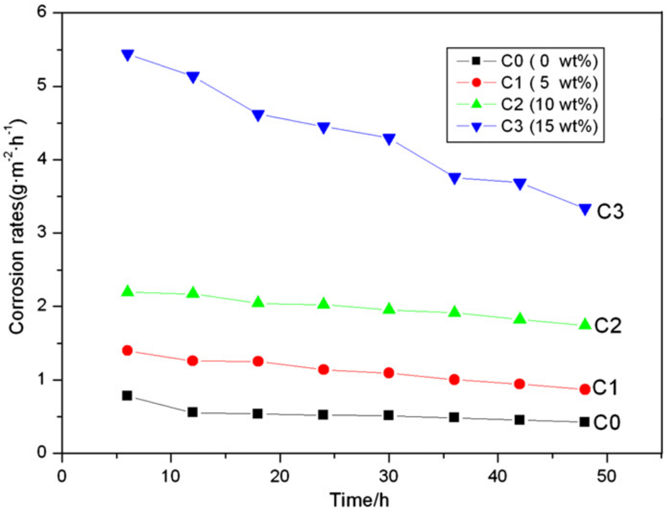

Fig. 2. Corrosion rates of cermets versus time in 2 mol/L HNO_3 solution.

Table 3
Properties and characteristics of cermets.

Cermets	Theoretical density (g cm^{-3})	Actual density (g cm^{-3})	Relative density (%)	Low magnification microstructure
C0	5.6058	5.5890	99.7	A02B00C00
C1	5.8291	5.8116	99.7	A02B00C00
C2	6.0710	6.0589	99.8	A02B00C00
C3	6.3338	6.3211	99.8	A02B00C00

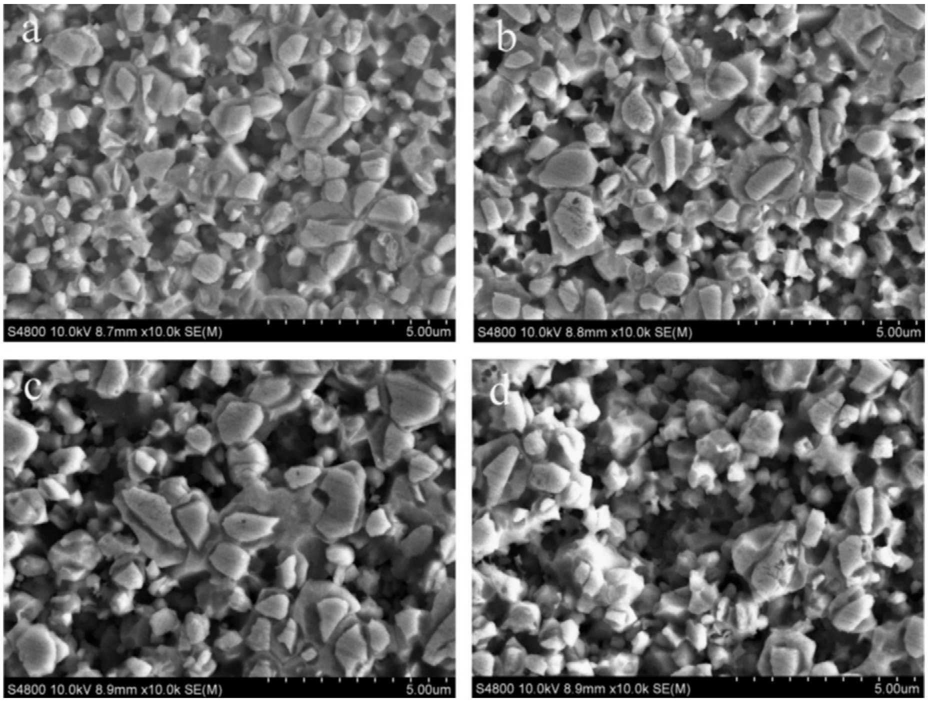

Fig. 3. SEM–SE micrographs of the corrosion surface of Ti(C, N)-based cermets immersed in 2 mol/L HNO_3 solution for 10 h: (a) C0: 0 wt% WC; (b) C1: 5 wt% WC; (c) C2: 10 wt% WC and (d) C3: 15 wt% WC.

phase skeleton is almost not observed in Fig. 3(d). It is observed that the remaining core/rim phase decreases remarkably after corrosion and small core particles exist at the attacked surface, as shown in Fig. 3(c). When WC content reaches up to 15 wt% in Fig. 3(d), the typical core–rim structure decreases greatly and a large number of core particles exist widely at the damaged surface. This indicates that both the inner rim phase and the outer rim phase are corroded and only the black core Ti (C, N) is left.

According to the corrosion morphology, in cermet C0, except the corrosion of Ni binder phase, the inner rim phase is also corroded seriously. The inner rim phase is (Ti, Mo) (C, N) solid solution and has a higher Mo content than the outer rim phase. Choi et al. [28] have reported that the corrosion resistance of cermets decreases when Mo is added. Hence, the corrosion resistance of (Ti, Mo) (C, N) solid solution will reduce with Mo addition.

According to relevant reports in the literature [26], WC content in the raw materials has no influence on the W content in the inner rim phase and W content is about 30 wt%. Different from the inner rim phase, W content in the outer rim phase will increase with WC addition in the raw materials when WC content is below 10 wt%. And when WC content exceeds 10 wt%, W content in the outer rim phase will be approximately 30 wt%, equal to that in the inner rim phase. Therefore, only the inner rim phase is observed and the outer rim phase is not observed in SEM micrographs sometimes. In cermet C1, W content in the inner rim phase is higher than that in the outer rim phase, hence the corrosion resistance of (W, Mo, Ti)(C, N) solid solution decreases with W content. Consequently, the degradation of the inner rim phase is significant, compared with the outer rim phase, as shown in Fig. 3(b). But when WC content reaches up to 10 wt% and 15 wt% in Fig. 3(c) and (d), W content in the outer rim phase is approximately equal to that in the inner rim phase. So the outer rim phase is also inferiorly resistant to corrosion and the residual grain is only black core particles at the damaged surface, especially in Fig. 3(d). It is well found that the degradation of outer rim phase is the most terrible in cermet C3 and a large number of core particles take the place of the core/rim structure in Fig. 3(d), only leaving very small core/rim structure at the attacked surface. In addition, owing to the increase of white core-grey rim structure, a large quantity of white core-grey rim structure is corroded completely. Thus, many corrosion pits exist and hard phase skeleton is damaged, as shown in Fig. 3(c) and (d). Moreover, a large number of unsupported hard phase particles fall off from the attacked surface. This is one of the reasons that the hard phase skeleton is damaged. From the above, it is well known that there exist two main reasons about the phenomenon that the corrosion rate increases with WC addition: one is that the corrosion resistance of rim phase decreases, especially the outer rim phase; the other is the unsupported hard phase particles fall off from the surface of cermets owing to the degradation of Ni binder phase.

The corrosion behavior of Ti(C, N)-based cermets has been explained by two main reasons. One is the dissolution of Ni binder phase, the other is the oxidation of carbides in the nitric acid solution.

Firstly, compared to carbides, Ni metal has strong reducibility and is easily oxidized based on the following reactions [13,14]:

$$3Ni+2HNO_3 = 3NiO+2NO\uparrow+H_2O \quad (2)$$

$$NiO+2H^+ = Ni^{2+}+H_2O \quad (3)$$

$$Ni+2H^+ = Ni^{2+}+H_2\uparrow \quad (4)$$

Owing to the degradation of Ni binder phase, the unsupported core/rim phase falls off from the attacked surface.

On the other hand, the oxidation of carbides includes WC, Mo_2C and even Ti(C, N). Due to the fact that the HNO_3 solution is a kind of oxidizing acid and the oxidability is the key factor of the corrosion of Ti(C, N)-based cermets. These reactions occur [8,15,29–31]:

$$3WC+10HNO_3 = 3WO_3+10NO\uparrow+3CO_2\uparrow+5H_2O \quad (5)$$

$$3Mo_2C+16HNO_3 = 6MoO_3+16NO\uparrow+3CO_2\uparrow+8H_2O \quad (6)$$

$$6Ti(C,N)+16HNO_3 = 6TiO_2+16NO\uparrow+6CO_2\uparrow+3N_2\uparrow+8H_2O \quad (7)$$

Both WO_3 and MoO_3 are unstable oxides which can react with the hydrogen ion as the following reactions:

$$WO_3+6H^+ = W^{6+}+3H_2O \quad (8)$$

$$MoO_3+6H^+ = Mo^{6+}+3H_2O \quad (9)$$

Therefore, the reactions (6) and (9) occur in the rim phase of cermets without WC addition. However, WC addition is more easily oxidized and dissolved in the nitric acid solution, compared with Ti (C, N). When WC is added in the cermets, the reactions (5) and (8) also happen in the rim phase and white core. Thus, the corrosion of cermets becomes more considerable compared with that of cermets without WC addition. Although W content in the inner rim phase is almost unchanged, W content in the outer rim phase increases when WC content increases in the raw materials. Hence, the corrosion reactions (5) and (8) further occur in the outer rim phase due to the increase of W content in the outer rim phase. As a result, both the rim phase and the white core are corroded, only leaving the black core particles at last. Additionally, the oxidation of Ti(C, N) is possible according to the thermodynamics condition and the final product TiO_2 which is an extremely stable oxide. Therefore, TiO_2 oxide film can prevent the reaction (7) further occurring and the black core particles are relatively intact in cermets.

Furthermore, according to the electrochemical theory, the micro batteries are formed and electrochemical reactions will happen owing to the potential difference between the two phases. The potential difference between Ti(C, N) and Ni is the highest in Ti(C, N)–Mo_2C–Ni system. Even though WC is added in the cermets, the conclusion is still applicable. So the corrosion of binder phase is more vulnerable compared with the rim phase. Although the galvanic couples with Ni as the anode are predominant, the galvanic couples Ti(C, N) (cathode)/Mo_2C (anode) and Ti(C, N) (cathode)/WC (anode) occur as well in the system. Thus, the inner rim phase is also attacked heavily. As WC content increases, the galvanic couple Ti(C, N) (cathode)/WC (anode) also occur severely in the outer rim phase and the white core-grey rim structure. As a result, the degradation of the whole rim phase and white core is also significant.

4. Conclusions

In this paper, the microstructures and corrosion resistance of Ti(C, N)-based cermets with different WC content in 2 mol/L HNO_3 solution are investigated. And the corrosion mechanism is discussed in detail. The conclusions are summarized as follows:

(1) The microstructures of Ti(C, N)-based cermets exhibit the black core-grey rim structure and white core-grey rim structure, and the rim has two layers including the inner rim and outer rim. The initial Ti (C, N) particles, which are not completely dissolved during sintering, normally appear as black cores. The white core, inner rim and outer rim are (Ti, W, Mo) (C, N) solid solution formed at different sintering stages. Compared with the outer rim and black core, however, the white core and inner rim contain higher Mo and W element and appear brighter since the elements with larger atomic number appear brighter in color in SEM.

(2) With the increase of WC content, although the thickness of the inner rim increases, the size of core/rim structure has a trend to become finer. Meanwhile, the amount of white cores increases and that of black cores decreases. WC can improve the wettability between Ti(C, N) hard phase and Ni binder phase and restrict the grain growth.

(3) The corrosion rates of all cermets decrease with the accumulation of immersion time. On the other hand, the corrosion rate of cermets increases with WC addition.

(4) In Ti(C, N)-based cermets system, Ni binder phase is the most vulnerable composition in HNO_3 environment. Some unsupported hard phase particles fall off from the attacked surface due to the degradation of binder phase. On the other hand, WC addition is more easily oxidized and dissolved in the nitric acid solution, compared with Ti (C, N) particles. Consequently, both the inner rim phase and the white core are severely corroded with the increase of WC addition. When the WC content exceeds 10 wt%, the degradation of the outer rim phase becomes considerable as well.

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