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Strength properties and fracture behavior of ZrC particle-reinforced tungsten composite ①

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[Abstract] The flexural strength of 30% (volume fraction) ZrC particle-reinforced tungsten composite (ZrC_p/W) at 20 ~ 1200 ℃ were measured using three-point bending, and the fracture behaviors of the samples at 20 ℃ and 1000 ℃ were studied with a scanning electron microscope. As temperature increases, the strength of the composite increases firstly and the highest strength value, 829 MPa, is measured at 1000 ℃, and then the strength decreases when temperature is over 1000 ℃. The fracture of the composite at 20 ℃, which is controlled by the crack initiating process, is brittle, and the corresponding stress-deflection curve is linear. There is a metastable growth and coalescence of the initial cracks during the fracture process of the samples at 1000 ℃, and the stress-deflection curve displays a nonlinear characteristic. The good elevated strength of the composite is partly attributed to the W grain interior strength and ZrC_p/W interface strength. The reinforcement at high temperature is mainly attributed to the load transfer of ZrC_p/W interface and dislocation strengthening.

[Key words] tungsten composite; zirconium carbide; strength; fracture behavior
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1 INTRODUCTION

Superior mechanical properties like high melting point, high density, good high temperature strength and high elastic modulus make tungsten as a kind of attractive materials for many important structural applications at high temperatures. But its strength is still decreases greatly with increasing temperature. For example, the strength of pure W material decreases by about 60% compared with that at room temperature[1]. In order to improve the high temperature strength of tungsten, refractory oxide and carbide phases, such as ThO_2[1], ZrO_2[2], Al_2O_3[3], HfC[4], ZrC[5], etc., are used as dispersive strengthening phases. Maybe due to the lower content of these reinforcements (usually less than 2%, volume fraction), the strength still drastically drops at high temperatures. The need of new materials having higher temperature capability compared to the conventional metals and alloys has led to the development of metal matrix composites. To date, the researches on tungsten-ceramic composites with high ceramics particles content are seldom reported.

In our previous work[6~9], tungsten matrix composite containing 20% (volume fraction) ZrC[6] or 30% (volume fraction) TiC[7] particles have excellent elevated temperature strength and ablation resistance[8], and they should be a good potential material for the aerospace and energy industry. However, the fracture behavior and strengthening mechanisms of ZrC_p/W composites at both room temperature and elevated temperature have not been sufficiently clarified yet. Tungsten is brittle at room temperature and ductile at high temperature, so the strength and fracture behavior and strengthening mechanisms at high temperatures should be different from that at room temperature. The purpose of the present work is to investigate the strength properties and fracture behavior of ZrC_p/W composite at various temperatures.

2 EXPERIMENTAL

Commercial tungsten powders with an average size of 3.5 μm were used as the matrix, and ZrC powders with an average size of 2.5 μm were used as the reinforcement. The starting mixed powders containing about 70% W and 30% ZrC were mixed for 24 h by ball milling. The mixtures were preformed in a steel die under 200 MPa, and then hot pressed at 2000 ℃, 20 MPa for 1 h in a vacuum of 1.3×10^{-3} Pa. To compare with the composite, similar conditions were used to prepare pure tungsten.

The specimens with dimension of 2.5 mm × 4 mm × 30 mm were used for flexural strength measurement by the three-point bending method with a

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20 mm span. Room temperature measurement was conducted in air, and elevated temperature (600 ~ 1 200 ℃) measurement was carried out in an argon atmosphere. Additionally, most of the bending tests were usually interrupted before failure for several times in order to observe the crack formation and crack propagation in ZrC_p/W specimens. The fracture surfaces and crack path in the specimen surfaces were examined with a scanning electron microscope.

3 RESULTS

Flexural strength of ZrC_p/W composite basically exhibits an increase trend over room temperature as test temperature increases from 20 to 1 200 ℃, as shown in Fig. 1. The highest strength value of 829 MPa appears at 1 000 ℃, an increase of 17.6% compared with the corresponding room temperature strength value of 705 MPa. Strength decreases with further increasing temperature from 1 000 to 1 200 ℃, but the strength value at 1 200 ℃ is 810 MPa, which is still higher than that at room temperature. On the contrary, the strength of pure W materials always decreases with increasing temperature. The specimen of the pure W materials breaks without plastic deformation at 600 ℃ and bends without fully fracture even if the cracks take place under the bend condition at 800 ℃, which implies that pure W material has a good ductility at and above 800 ℃ and 800 ℃ is above the ductile-brittle transition temperature.

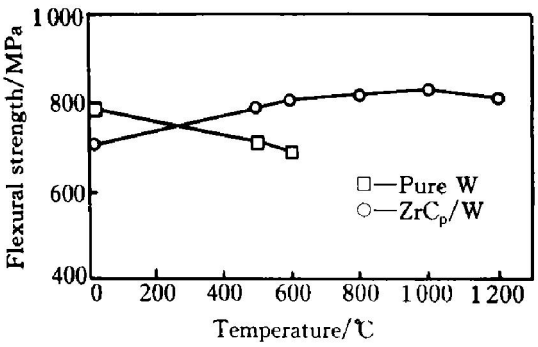

Fig. 1 Variation of flexural strength of ZrC_p/W composite and pure W with temperature

The stress-deflection curves of ZrC_p/W composite at different temperatures are plotted in Fig. 2. The curve tested at 20 ℃ indicates that there is only an elastic deformation in ZrC_p/W specimen before failure, but the curves tested at 1 000 ℃ and 1 200 ℃ show non-linearity before failure, which implies that there should be a plastic deformation and/or a metastable crack propagation before failure in the composite. The general trend is that the flexural strength increases and the deflection (or strain) increases with increasing temperature, and a little of plastic deformation takes place before failure at high temperature.

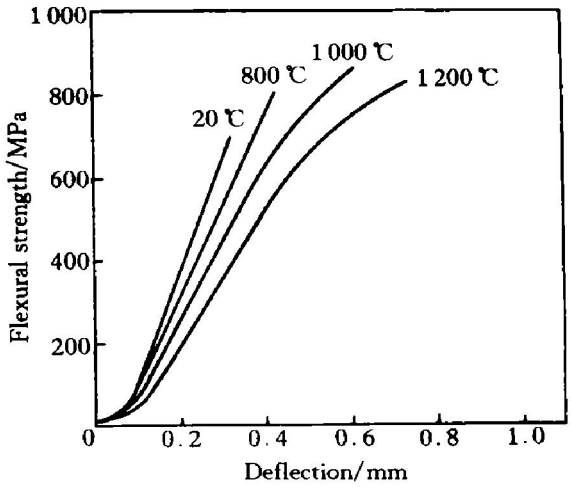

Fig. 2 Stress-deflection curves of ZrC_p/W composite at various temperature showing a little of plastic deformation taken place at 1 000 ℃ and 1 200 ℃

The SEM micrographs of the fractograph of ZrC_p/W and pure W specimens tested at various temperatures are shown in Fig. 3. The fractograph of pure W tested at room temperature shows a typical brittle fracture mix mode of intergranular and transgranular fracture (Fig. 3(a)), and the fracture at 600 ℃ is intergranular (Fig. 3(b)). The morphological features of the fractograph of W matrix in ZrC_p/W composites (Fig. 3(c)) are similar to that of pure W materials (Fig. 3(a)) tested at room temperature. However, the size of W grains in ZrC_p/W composite is significant smaller than that of pure W materials, which implies that ZrC inhibited the growth of W grain during the sintering process. On the fractograph of ZrC_p/W composite tested at 800 ℃, W matrix still exhibits a mix fracture mode of intergranular and transgranular fracture (Fig. 3(d)). On the fractograph of ZrC_p/W tested at 1 000 ℃ (Fig. 3(e)), some fine tear ridges can be observed, indicating a plastic deformation of W matrix, and the similar phenomenon can be found on the fractographs tested at 1 200 ℃ (Fig. 3(f)). On the other hand, a lot of transgranular fractures of W grains in W matrix are observed not only at 1 000 ℃ but also at 1 200 ℃, which is not similar to the fracture of pure W material tested at and above 600 ℃ where no transgranular fracture of W matrix is found except the intergranular fracture of W matrix.

Fig. 4 shows a crack in a failed ZrC_p/W specimen tested at room temperature. Although the crack deflection evidence can be found, the crack deflection is not significant. The specimen ruptures in a brittle mode at room temperature, and its stress/deflection curve is linear(which is marked by 20 ℃, as shown in Fig. 2).

The dynamic crack observation of the tensile surface of ZrC_p/W composite during bending at 1 000 ℃ was carried out by interrupting the bend test process

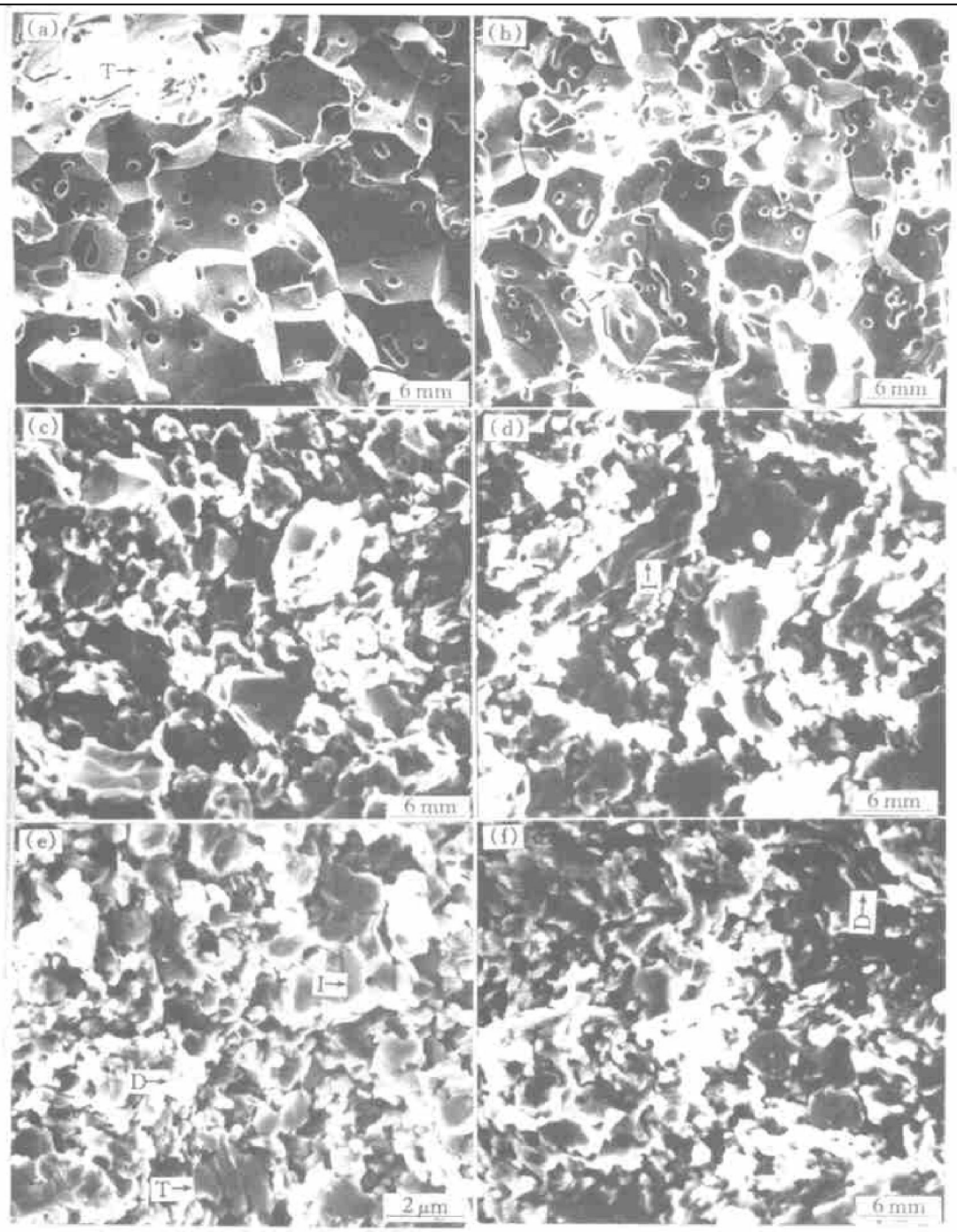

Fig. 3 Fractographs of ZrC_p/W composite and pure W material tested at various temperatures
(a) —Pure W, room temperature; (b) —Pure W, 600 ℃;
(c) —ZrC_p/W, room temperature; (d) —ZrC_p/W, 800 ℃; (e) —ZrC_p/W, 1 000 ℃;
(f) —ZrC_p/W, 1 200 ℃, showing transgranular fracture (T), intergranular fracture (I) and fine dimples (D) of W matrix

for several times. Fig. 5(a) shows some microcracks initiated in the bigger ZrC particles, ZrC clusters or W matrix on the tensile surface. Increasing the applied load, the crack deflects the ZrC particle and then propagates in tungsten matrix, or the ZrC particle arrests the crack when the crack meets with a ZrC particle, and the separation of ZrC_p/W interfaces is also observed, as indicated by arrows in Fig. 5(b). The phenomenon of interface crack is seldom found in this study. When the load is further increased, the microcracks grow to form discontinuous cracks of 10 ~ 30 μm in length, resulting in the formation of bigger cracks (macrocracks) (Fig. 5(c)). During the coalescence of short cracks, ductile tears in W matrix usually can be observed (see Fig. 5(d)). In fact, Fig. 5(a) ~ (d) give out the crack growth insitu in the ZrC_p/W specimen tested at 1 000 ℃.

4 DISCUSSION

The tungsten is brittle at room temperature and ductile at high temperature[10]. When a crack initiates in ZrC_p/W composite at room temperature, the brittle ZrC and W matrix can not effectively prevent

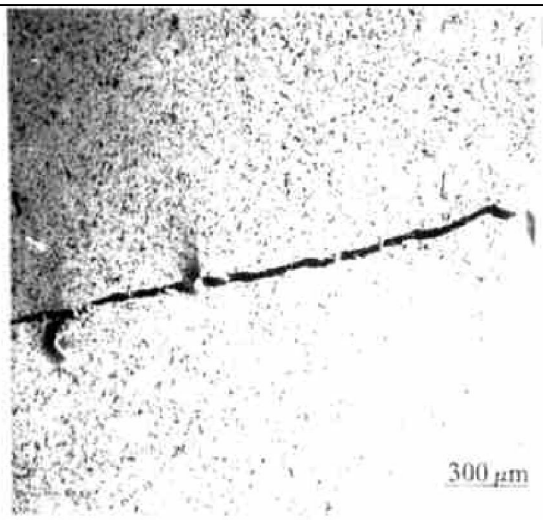

Fig. 4 Crack in ZrC_p/W specimen failed at room temperature

the crack propagating due to the lack of ductility, which results in a brittle fracture under a relative lower load. Consequently, the room temperature strength of ZrC_p/W composite is lower. The pure W specimen bends without fracture under three-point bending condition of 800 ℃, which reveals that W has good plasticity at and above 800 ℃.

At elevated temperature (for example, 1 000 ℃), W matrix obtains its ductility by grain boundary sliding and/or dislocation sliding. If a crack is initiated in a ductile W matrix, it can be governed by the ductility of W. The ZrC_p/W interfaces or ZrC particles in front of the initial microcracks maybe cracks, but the propagating cracks can be arrested by the ductile W matrix. The facts of microcracks formation, growth and coalescence, and ductile tearing in W matrix (Fig. 5) show that the ZrC_p/W composite can withstand greater damage at elevated temperature than at room temperature. The corresponding stress/deflection curves at 1 000 ℃ and 1 200 ℃ also confirm that. On the other hand, ZrC hard particles are presented in the ductile W matrix, the particles and the particle-introduced misfit stress field can strongly constrain the plastic deformation of W matrix by hindering the W grain boundaries sliding and dislocations motion. Consequently, enhance the elevated temperature strength of ZrC_p/W composite.

The fracture behavior observation (Fig. 5) clearly demonstrates that the bonding strength of ZrC_p/W interface should be higher than that of W matrix. Additionally, the modulus of ZrC is 450 GPa[11], which is higher than that of W (385 GPa, when the

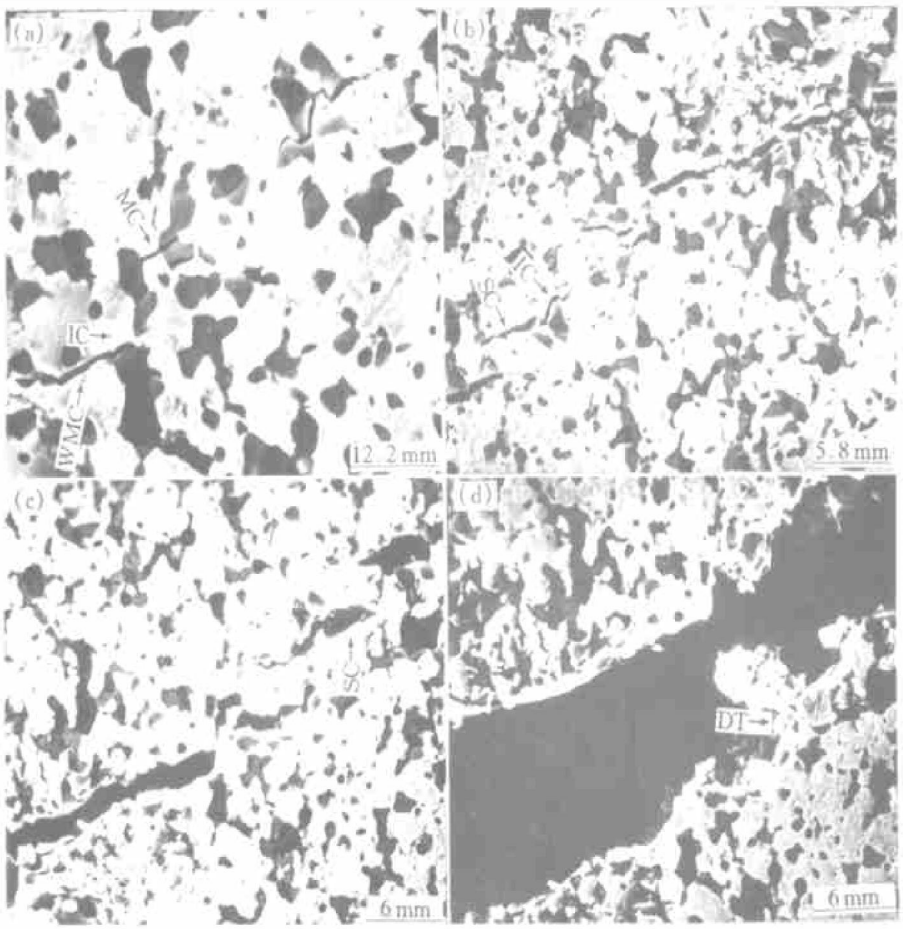

Fig. 5 Cracks in ZrC_p/W specimen tested at 1 000 ℃

(a) —Microcracks; (b) —Short cracks; (c) —Crack growth and coalescence; (d) —Ductile tear showing various fracture modes, such as cleavage of ZrC particle or ZrC cluster (PC), W matrix crack (WMC), ZrC_p/W interface crack (IC), microcrack (MC) and ductile tear (DT); SC standards for short crack

relative density of pure W is 100%[1]), therefore, ZrC particles carry more stress than W matrix when they are deformed to the same strain. Consequently, load transfer of ZrC_p/W interface is an important strengthening mechanism.

The damage modes of pure W material and ZrC_p/W composite at elevated temperature are illustrated in Fig. 6. Fig. 3(b) reveals that the pure W tested at 600 ℃ fractures in an intergranluar mode, however, both of transgranular and intergranular fractures are found in the fracture surface of ZrC_p/W composite tested in the range of 800~ 1 200 ℃. The large decrease of the flexural strength above 600 ℃ for the pure W is due to intergranular fracture. The fact that the ZrC_p/W composite has a higher elevated flexural strength than pure W is partly attributed to the difference in the fracture mechanism. The intergranular fracture is limited by the structure of ZrC_p/W composite. At elevated temperature, grain boundaries often act as sources of weakness because of grain boundary sliding. Therefore it is important to limit grain boundary sliding in order to attain a decent strength at elevated temperature[12]. When ZrC particles are added into W matrix, ZrC_p/W interfaces will be formed. Due to the higher strength of ZrC_p/W interface compared with that of W grain interior, a lot of transgranular fractures of W grains appear at elevated temperatures, as shown in Fig. 3. Thereby, with the attribution of W grain interior strength and ZrC_p/W interface strength at elevated temperature, the strength of the composite is improved.

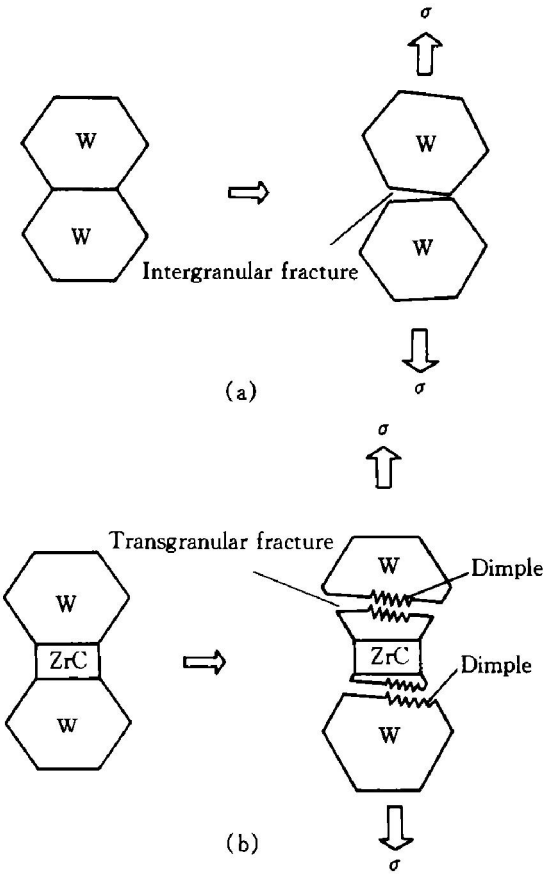

Fig. 6 Damage modes of pure W and ZrC_p/W composite at elevated temperature
(a) —Pure W; (b) —ZrC_p/W

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