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Wear





Friction and wear properties of in situ (TiB₂ + TiC)/Ti₃SiC₂ composites

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ABSTRACT

The non-lubricated, ball-on-flat sliding friction and wear properties of in situ (TiB $_2$ +TiC)/Ti $_3$ SiC $_2$ composites against bearing steel ball were investigated. The load was varied from 10 to 30 N. The material without TiB $_2$ shows an increasing friction coefficient with the increasing load. However, for (TiB $_2$ +TiC)/Ti $_3$ SiC $_2$ composites, the friction coefficient shows an insensitivity to load. Both friction coefficient and wear rate initially increase and then dramatically decrease with TiB $_2$ content increasing from 0 to 20 vol%. (TiB $_2$ +TiC)/Ti $_3$ SiC $_2$ composites with 15 vol% and 20 vol% TiB $_2$ show excellent wear resistance, whose friction coefficient and wear rate are much lower than those of TiC/Ti $_3$ SiC $_2$ composite. The enhanced wear resistance is mainly attributed to the facts that the hard TiB $_2$ and TiC particles nail the surrounding soft matrix and decentralize the shear stresses under the sliding ball, a self-lubricating oxide debris forms on the wear surface, and the wear mode converts from adhesive wear to abrasive wear.

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1. Introduction

Ti₃SiC₂ is a representative among MAX phases, which combines merits of metals and ceramics, such as low density, high modulus, good thermal and electrical conductivity, excellent thermal shock resistance and high temperature strength, damage tolerance and easy machinability [1-3]. Therefore it is a promising candidate for new high temperature structural materials, electrode materials in molten metal, self-lubricating materials and electric brush materials. However, its low hardness, creep strength, oxidation and wear resistance restrict the potential application of Ti₃SiC₂. Incorporation of second phase is an effective way to overcome these weaknesses. A number of reinforcing agents including TiC, SiC, Al₂O₃, c-BN, TiB₂ and ZrO₂ have been applied to improve the mechanical properties of Ti₃SiC₂ [4-9]. However, previous studies mainly concentrated on the composites containing only one reinforcing agent and limited enhancement effect. In recent years, cooperative mechanism between two or more strengthening and toughening methods has received much attention. By this cooperative mechanism, the enhancement and toughening effect was usually more significant than the additional effect brought by the individual toughening mechanisms [10,11]. The various wellknown strengthening and toughening mechanisms could be used in cooperative toughening design of ceramics by appropriate methods.

Both TiB2 and TiC have high hardness, high modulus, excellent chemical stability, good electrical and thermal conductivity, and approximate coefficient of thermal expansion with Ti₃SiC₂. Therefore they are ideal candidate reinforcements for Ti₃SiC₂ matrix. Comparing to traditional preparation process, in situ synthesis shows many advantages in fabrication of composite ceramics, such as uniform distribution and fine grain size of reinforcements, clean and tightly bonded grain boundary, simplified process and excellent properties. Recently, Taimatsu et al. [12] reported the TiB₂-TiC-Ti₃SiC₂ composites which were synthesized by reactive hot pressing. In our previous work [13] (TiB₂ + TiC)/Ti₃SiC₂ composites have been successfully prepared by in situ hot-pressing sintering, in which Ti₃SiC₂ matrix is cooperatively reinforced by columnar TiB₂ and equiaxed TiC grains. Owing to the cooperative effect of the strengthening and toughening mechanisms such as particulate reinforcement, crack deflection, grain's pull-out and fine-grain toughening caused by the two reinforcing agents, the (TiB₂ + TiC)/Ti₃SiC₂ composites show excellent mechanical properties (bending strength, toughness and hardness).

Friction characteristics and wear resistance are two important properties that need to be characterized for Ti₃SiC₂-based materials as candidates for structural components. Many works have been carried out to investigate the tribology of pure Ti₃SiC₂ and Ti₃SiC₂-based ceramics [4,14–19]. In this paper, the reciprocating friction and wear properties of in situ (TiB₂+TiC)/Ti₃SiC₂ composites were systematically investigated and the effects of TiB₂ content and different loads under non-lubricated conditions were examined using a ball on flat sliding block, point contact wear mode.

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2. Experimental procedure

2.1. Materials preparation and characterization

TiH₂ (99.9%, 300 mesh), Si (99.9%, 300 mesh), graphite (99.9%, 200 mesh), and B₄C (99%, average particle size 3.5 μ m) were used as starting materials. In situ (TiB₂ + TiC)/Ti₃SiC₂ composites were prepared according to the general reaction as follows:

$$(3 + 3x + y)\text{TiH}_2 + \text{Si} + (2 + y)C + xB_4C \rightarrow \text{Ti}_3\text{SiC}_2 + 2x\text{TiB}_2 + (x + y)\text{TiC} + (3 + 3x + y)H_2 \uparrow$$
 (1)

The volume fraction of TiC in the composites was fixed at 10 vol% and the composites with different content of TiB $_2$ were denoted as TC (0 vol%) TC/5B (5 vol%), TC/10B (10 vol%), TC/15B (15 vol%) and TC/20B (20%), respectively. Excess Si (1.2 mol of Si) was added to insure the obtained TiC content in (TiB $_2$ +TiC)/Ti $_3$ SiC $_2$ composites close to theoretical one. After being mixed and dried, the mixtures were heated at $900\,^{\circ}\text{C}$ for 1 h and then hot pressed at $1500\,^{\circ}\text{C}$ for 2 h under an Ar atmosphere at $25\,\text{MPa}$.

The density of the as-prepared materials was determined by the Archimedes method. The phase compositions were examined by X-ray diffractometer (ARL XTRA, Switzerland). The microstructures were investigated by scanning electron microscope (SEM). Room temperature bending strength and fracture toughness were measured using three point bending method and single-edge notch beam (SENB) method respectively with specimen dimensions of $4\,\mathrm{mm} \times 3\,\mathrm{mm} \times 40\,\mathrm{mm}$. The Vickers hardness was measured by a microhardness tester at a load of 9.8 N with a dwell time of 10 s.

2.2. Wear test and characteristic

Friction and wear tests were performed in a UMT-2 multifunction test system (CETR, USA). A ball sliding on a linear reciprocating athletic flat specimen $(3 \text{ mm} \times 5 \text{ mm} \times 8 \text{ mm})$ was adopted. The specimens were cut by electrical discharge method and the test surface was mechanically polished down to 1200# SiC paper. Before and after testing, all samples were ultrasonically cleaned, dried, and weighed. The ball (φ 4 mm in diameter, made of AISI-52 100 bearing steel, with a density of 7.85 g cm⁻³ and Vickers hardness of 7.6 GPa) rubbed the surfaces (5 mm \times 8 mm) of the samples. The wear tests were performed under the loads of 10, 20, and 30 N with a constant stroke length of 4.50 mm at a frequency of 6.7 Hz. The total sliding distance was 36 m at a sliding velocity of 30 mm s⁻¹ for 1200s. All of the wear tests were conducted at room temperature in air with a relative humidity of 40-55%. Friction coefficient was measured by an analog-to-digital converter and recorded in a computer. The wear rate was calculated by

$$W = \frac{V}{PL} \tag{2}$$

where $V(\text{mm}^3)$ is the wear volumes obtained by measuring the weight loss in a microbalance (accuracy: 10^{-4} g) after ultrasonic cleaning and drying, and from the measured material density, P(N) is the applied load and L(m) is the total sliding distance. Three tests were repeated for each material and experimental condition. After tests, the worn surfaces and the collected wear debris were investigated by scanning electron microscope equipped with an energy-dispersive spectroscopy(EDS) system.



Samples	TC	TC/5B	TC/10B	TC/15B	TC/20B
Apparent porosity (%)	0.085	0.045	0.124	0.128	0.042
Bending strength (MPa)	415	475	700	666	495
Fracture toughness (MPa m ^{1/2})	7.79	9.08	9.55	8.04	7.47
Vickers hardness (GPa)	5.41	6.13	7.47	10.27	11.5

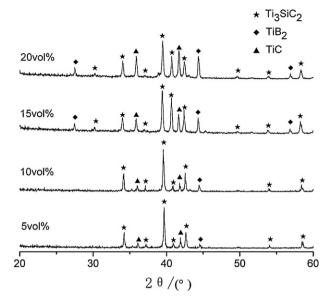


Fig. 1. XRD patterns of the materials with different TiB₂ content.

3. Results and discussion

3.1. Phase composition, microstructure, and mechanical properties

Fig. 1 shows the XRD patterns of the as-prepared in situ (TiB₂ + TiC)/Ti₃SiC₂ composites with different TiB₂ content. All the samples show the same phase composition, i.e., Ti₃SiC₂ is the main phase, TiB2 and TiC are the second phases. With the increase of TiB₂ content, peak intensity of TiB₂ increases. Fig. 2 shows the microstructure of fracture surface for in situ (TiB₂ + TiC)/Ti₃SiC₂ composites. Laminar Ti₃SiC₂ grains, columnar TiB₂ grains and equiaxed TiC grains were clearly identified. With the increment of TiB₂ content, the grain size of Ti₃SiC₂ matrix tends to decrease, which suggests that in situ incorporation of TiB2 particles effectively hinders the grain growth of Ti₃SiC₂ matrix. Table 1 lists the typical physical and mechanical properties of in situ (TiB₂ + TiC)/Ti₃SiC₂ composites. It can be seen that both bending strength and fracture toughness of the composites increase firstly and then decrease, which reach the maximum of 731 MPa and 9.35 MPa m^{1/2}, respectively at 10 vol% TiB₂. The Vickers hardness increases markedly from 6 GPa to 11.5 GPa.

3.2. Friction coefficient and wear rate

Figs. 3–5 show the friction coefficients versus sliding time curves of the composites with different ${\rm TiB_2}$ content under constant loads of 10 N, 20 N and 30 N, respectively. With increasing sliding time, the friction coefficients of most samples start at a relatively low value and increase to a steady high value after a short time. Whereas for TC at 20 N and TC/20B at 30 N, the friction coefficients reach the steady state with more time than other samples and several fluctuations are seen in the break-in period for the latter. Unlike these cases, the friction coefficient of TC at 10 N load gradually declines

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